

# Absolute Specular Reflectance Measurements at Angles and Beyond

MARK FISHER  
APPLICATIONS ENGINEER  
AGILENT TECHNOLOGIES

20 APRIL 2017



Agilent Technologies

## – What We Will be Going Over –

What are My Choices in Accessories ?

Are there Advantages/Disadvantages I Need to Know ?

What Kind of Measurements Can I Make?

Are there any Limitations ?

How Can I Make Absolute Specular Reflectance Measurements at the Angle of My Choosing?

Examples of Sample Measurements

Wrap Up and Question Time



# Thank the People That Got Us Here



FIFTH CONFERENCE PARTICIPANTS 1927

A. PICCARD, E. HENRIOT, P. EHRENFEST, E. HERZEN, TH. DE DONDER,  
E. SCHRÖDINGER, J.E. VERSCHAFFELT, W. PAULI, W. HEISENBERG, R.H. FOWLER,  
L. BRILLOUIN; P. DEBYE, M. KNUDSEN, W.L. BRAGG, H.A. KRAMERS, P.A.M. DIRAC,  
A.H. COMPTON, L. DE BROGLIE, M. BORN, N. BOHR, I. LANGMUIR, M. PLANCK,  
M. SKŁODOWSKA-CURIE, H.A. LORENTZ, A. EINSTEIN, P. LANGEVIN, CH.-E. GUYE,  
C.T.R. WILSON, O.W. RICHARDSON



# – Specular Reflectance –

How has it Been Done in the Past?

Fixed Angle

Relative Reflectance

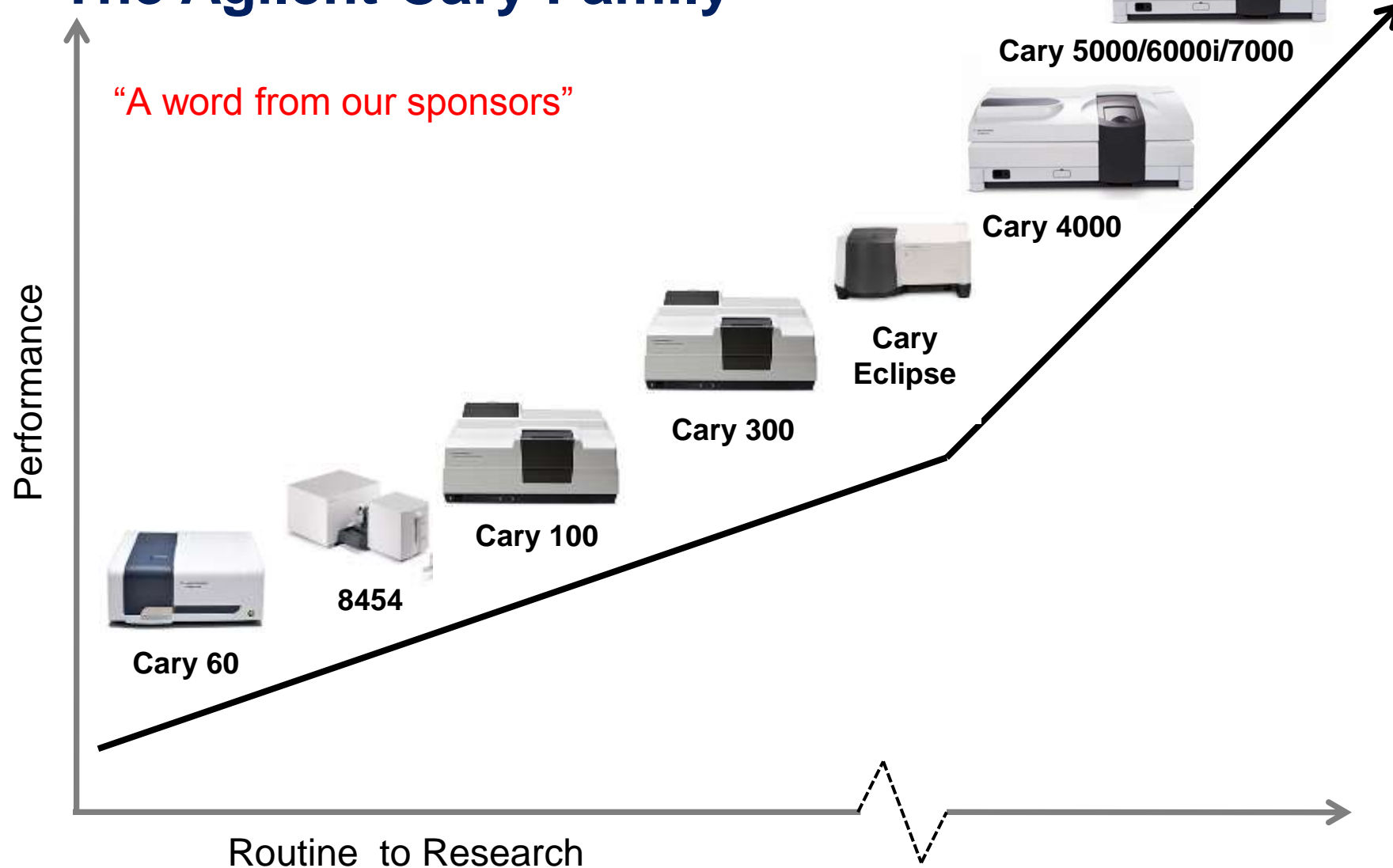
Absolute V-N V-W

Variable Angle



# UV-Vis NIR Spectroscopy Solutions

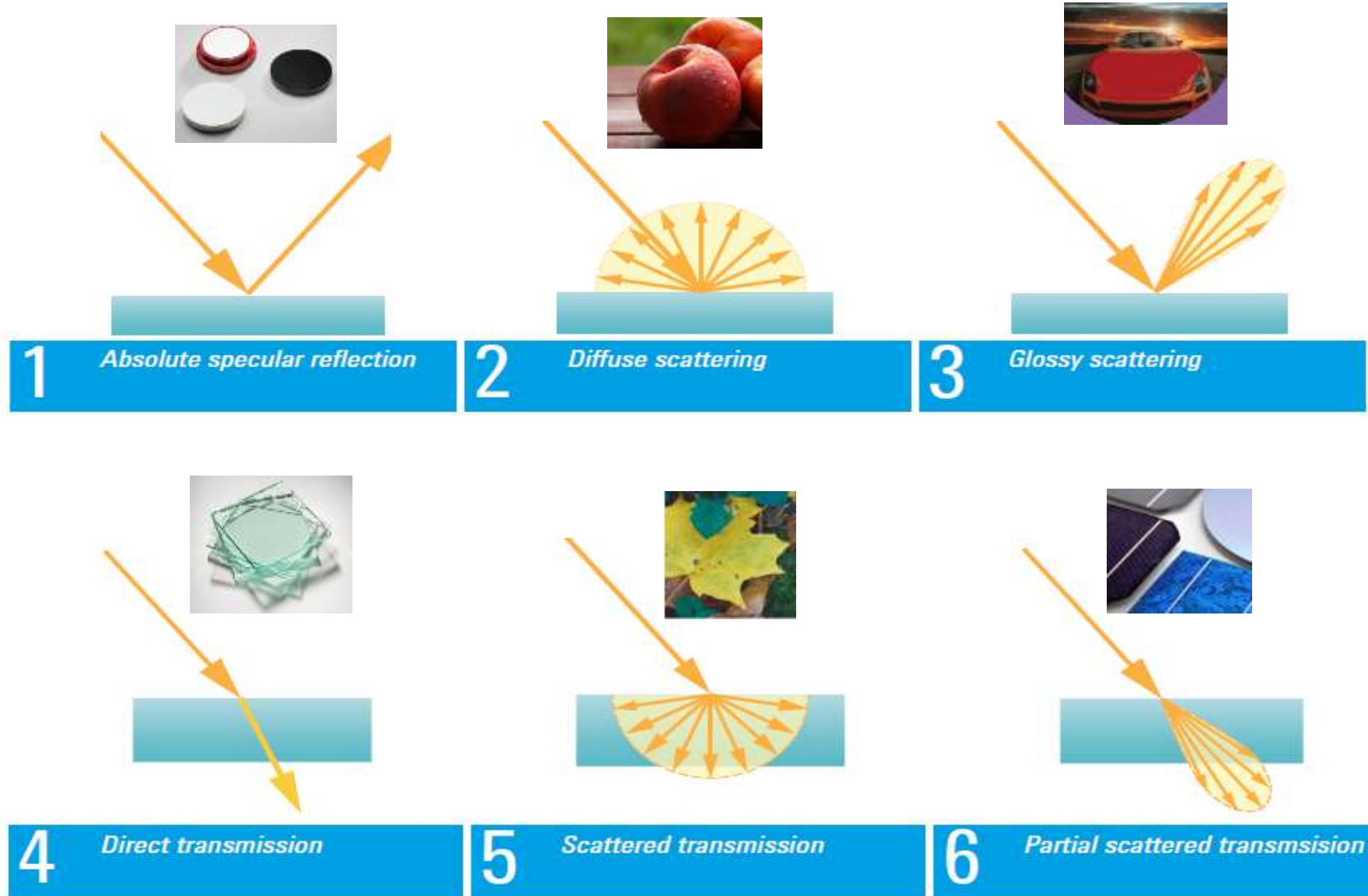
## The Agilent Cary Family



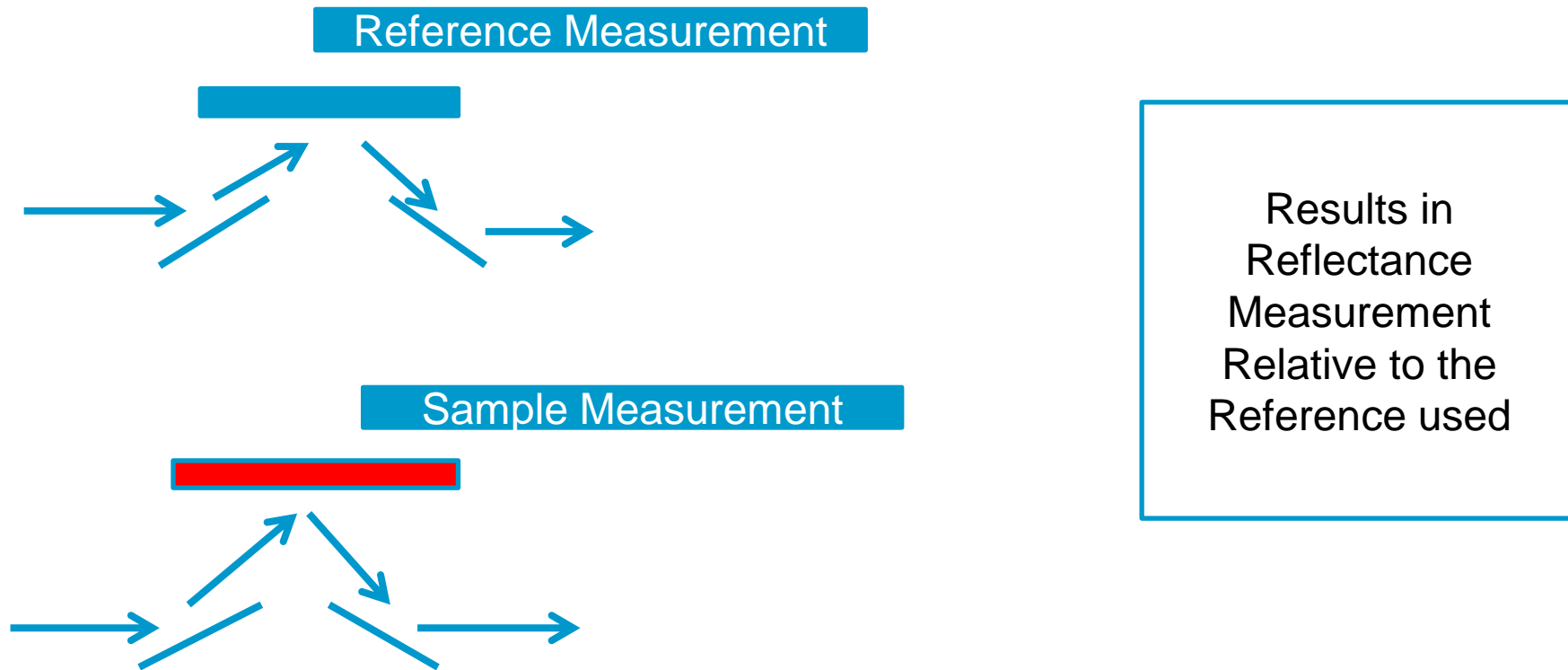
Agilent Technologies



# Measurement Modes



# Relative Reflectance Measurement

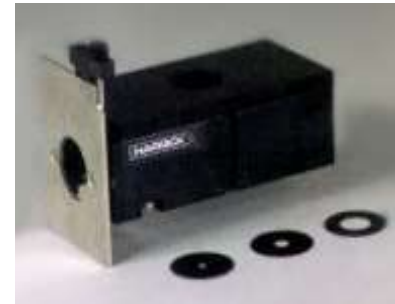


# Various Specular Reflectance Accessories



Variable Angle Specular Reflectance Accessory  
VASRA

20-70 deg (0.5 deg steps)



Fixed Angle Accessory



Absolute Specular Reflectance  
Accessory (V-W design)



Near Normal  
15 degrees  
30 degrees  
45 degrees



# Cary 7000 UMS and UMA

**U**niversal  
**M**easurement  
**A**ccessory



# Cary 7000 UMS and UMA



# Cary 7000 UMS and UMA

Cary 7000 **U**niversal **M**easurement **S**pectrophotometer



## Overview

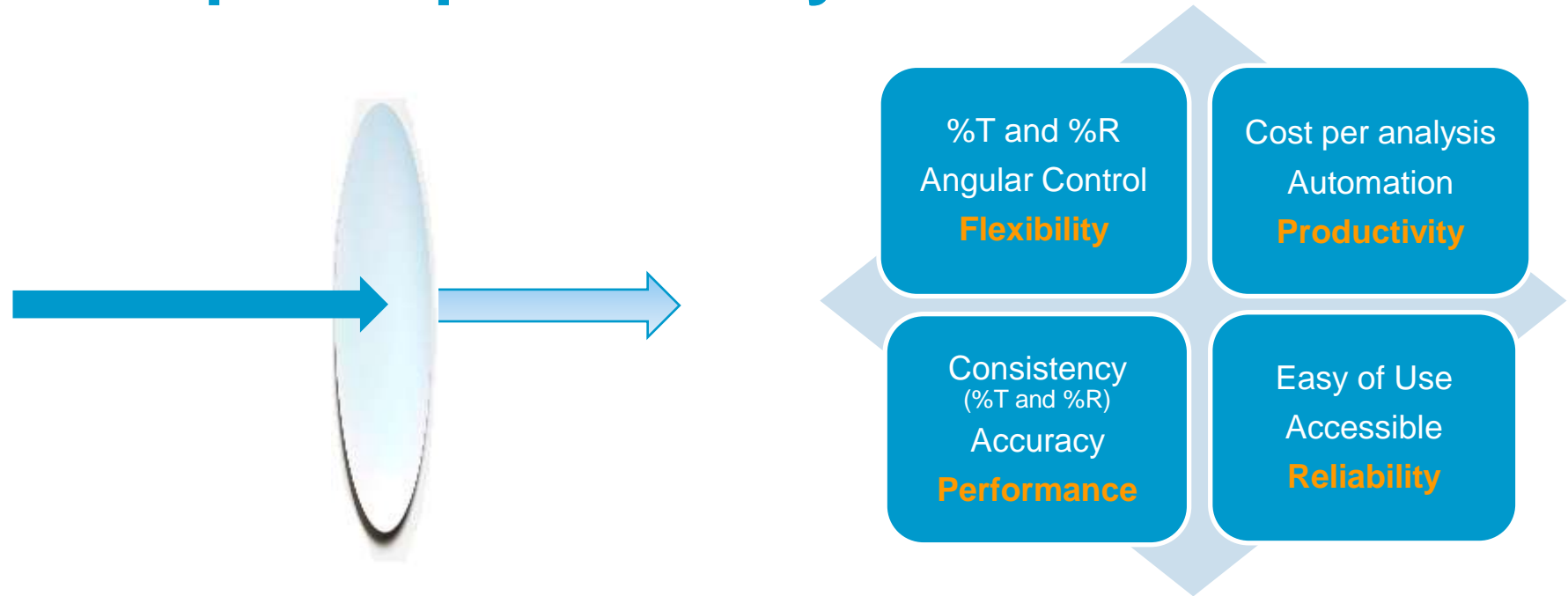
A turn-key, automated, **UV-Vis-NIR** spectrophotometer with Universal Measurement Accessory provides exceptional value, ease of use and performance – unmatched individually, unbeatable in a package.

## Application Focus (Solids)

- Photonics/Optics/Thin Films
- Material research
- Semiconductor
- Military/Defense
- Chemical/Industrial



# UV-Vis-NIR Spectrophotometry



Research



QA/QC Testing



Trouble Shooting





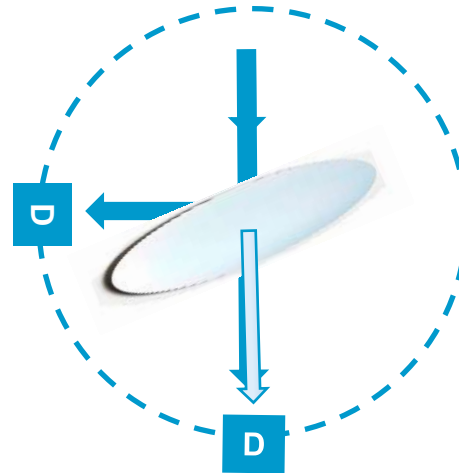
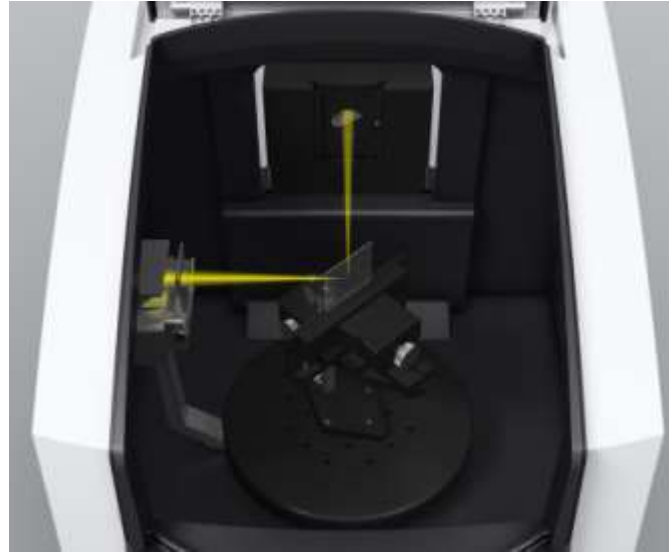
# Cary UMS Schematic

## Productivity

Automated *independent* control of polarization (s or p) detector (D) position and sample rotation.

*One baseline* is needed for all %R and %T measurements, at all angles for a given polarization – dramatically reducing total collect time.

Perform all %R, %T measurements on a single system eliminating accessory change over, or reconfiguration time.



## Performance

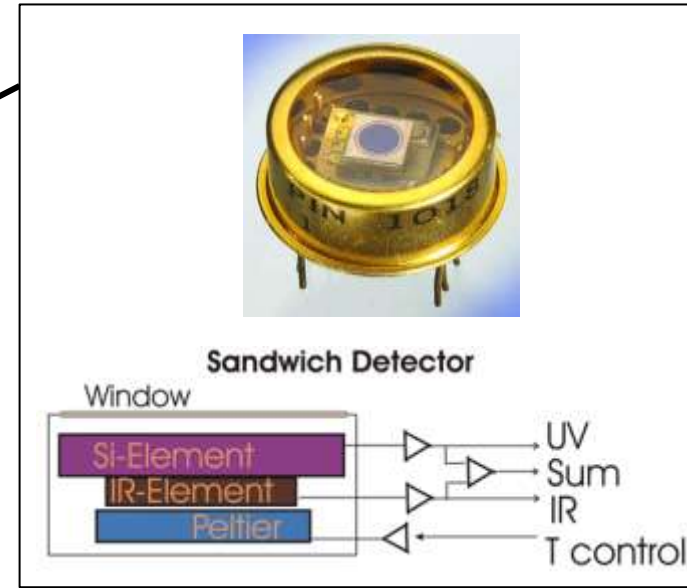
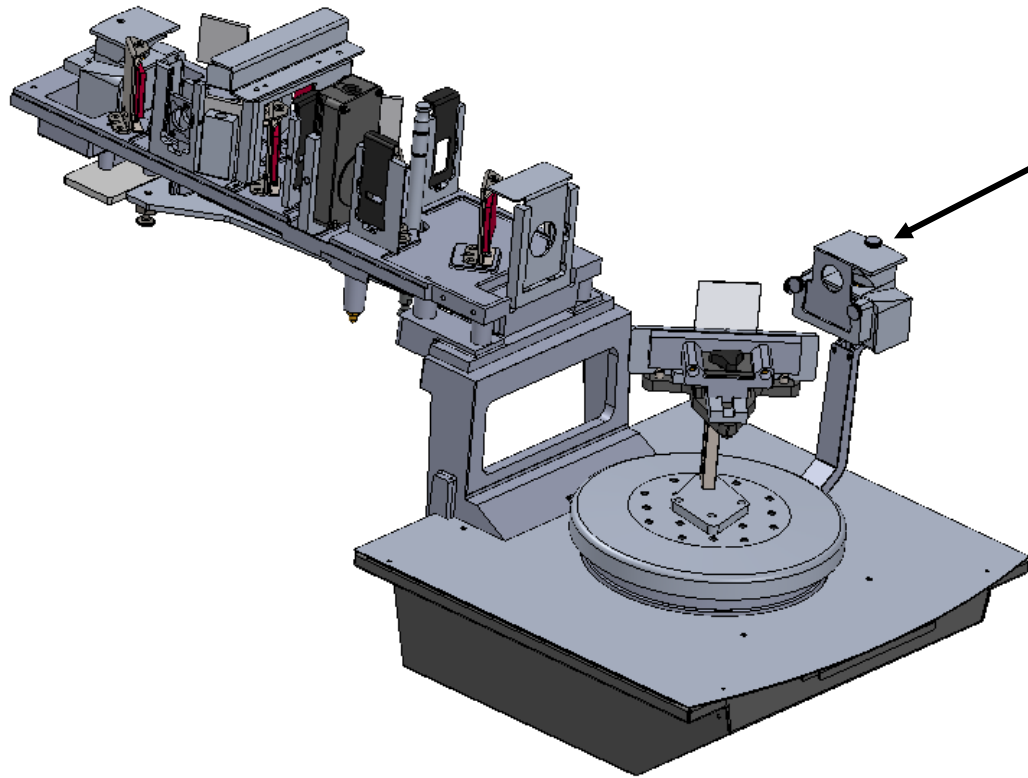
**Absolute reflection** and transmission by definition – the only difference between baseline and measurement is the sample itself.

Incident light is fixed in shape, and position, at the sample ensuring %T and %R are collected from the **same point on the sample**.

The detector has a pure line of sight of the sample. This unique **Direct View** provides the highest signal-to-noise improving accuracy, reproducibility and productivity



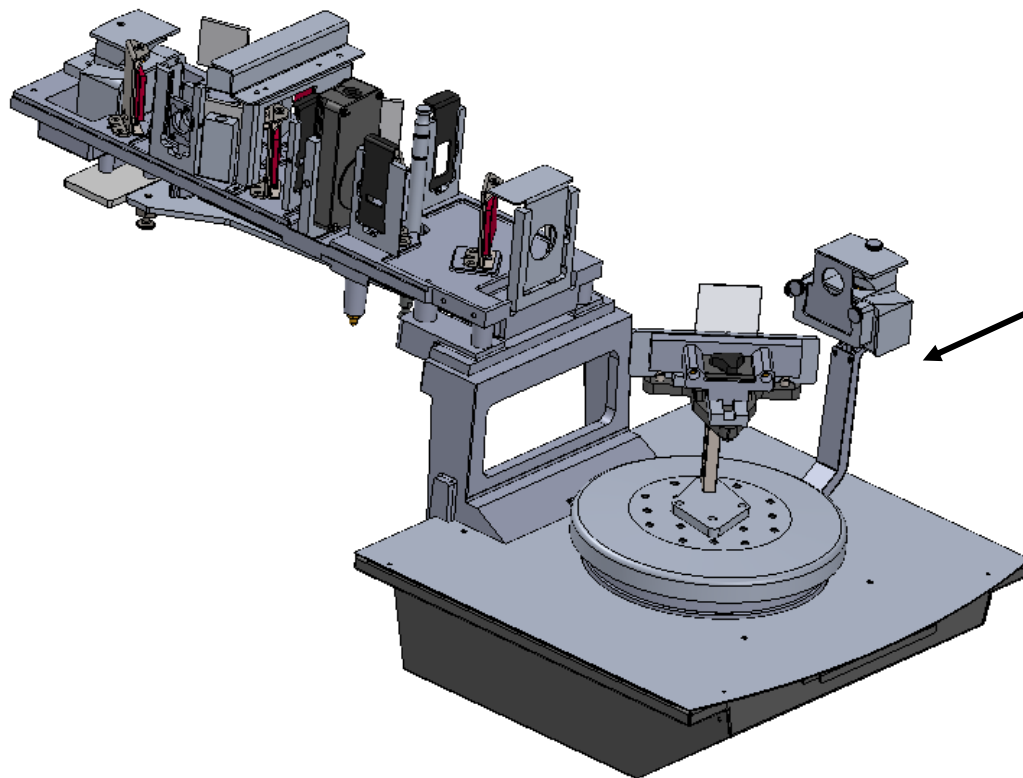
# Sandwich Detector



Feature	Advantage	Benefit
Si/InGaAs Sandwich Detector	Combined UV-Vis and NIR detector. No moving parts, ie. no beam shift at detector changeover.	Consistent data. Large wavelength range. (UV-Vis-NIR)



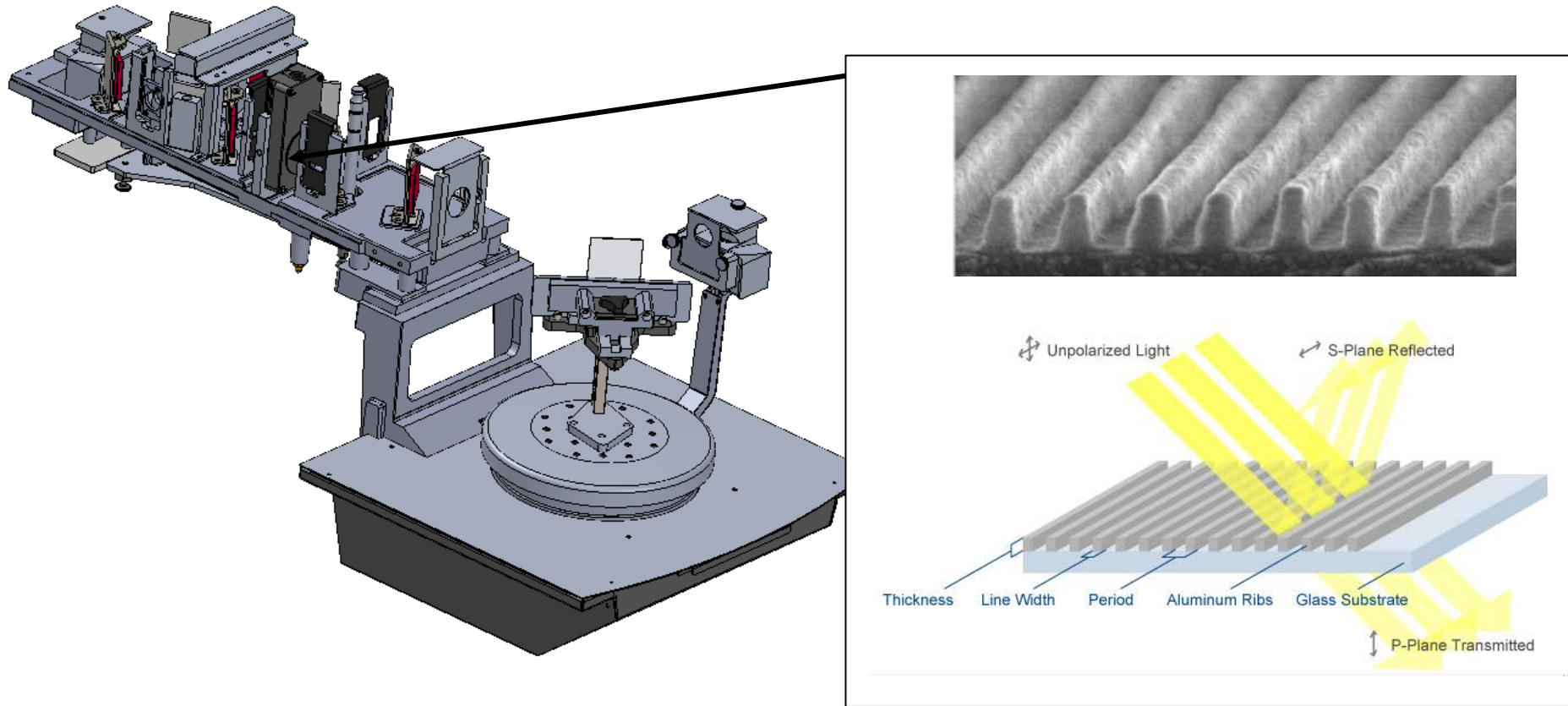
# Direct Detector Illumination



Feature	Advantage	Benefit
Direct Detector Illumination	All reflective, high efficiency, aluminum optics maintain high signal level and signal quality from source to detector  <b>Alternative</b> is using small DRAs and/or fiber optic/light pipes.	Productivity – saves time  <b>Loss in sensitivity, worse spectra quality or longer acquisition times.</b>

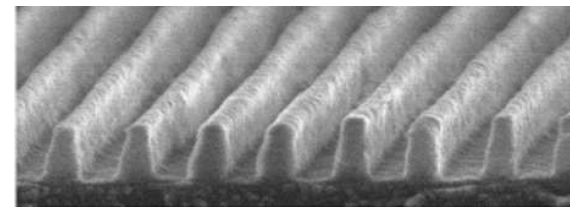
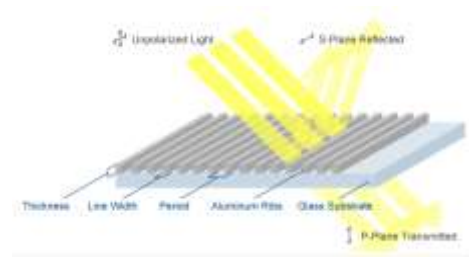


# Unique Wire Grid Polarizers



The Agilent Cary UV-Vis-NIR **automated polarizer** provides the next-generation of polarization control: A nano-wire grid which is lithographically laid onto a highly transmissive quartz substrate.

# Wire Grid Polarizer

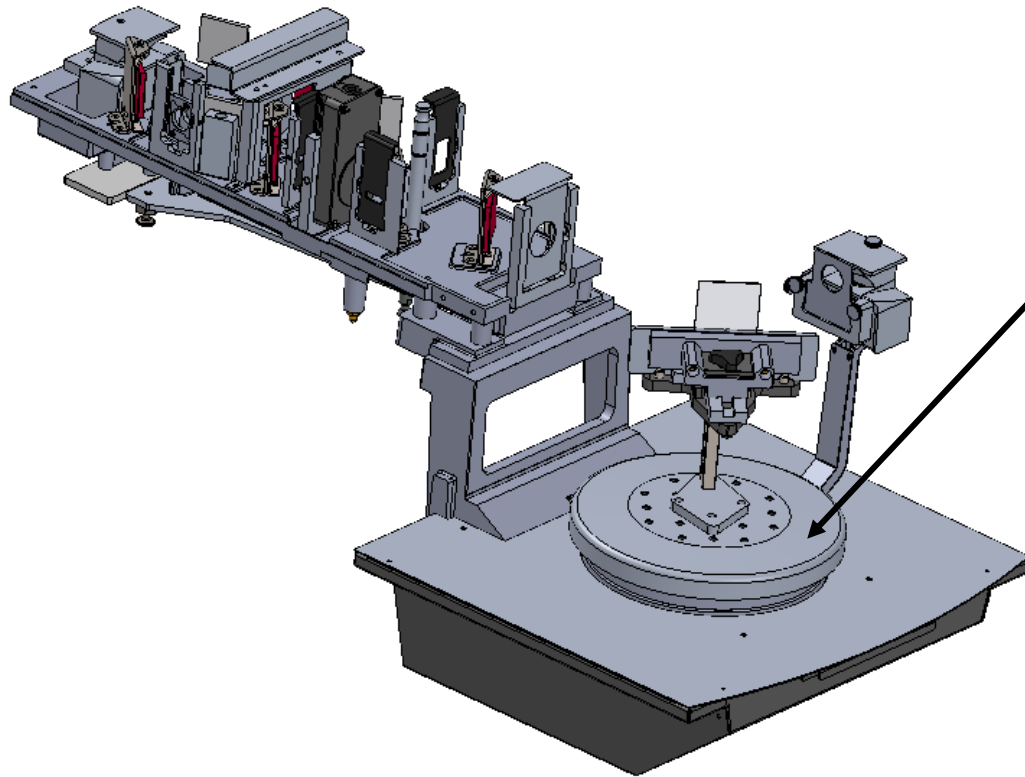


Feature	Advantage	Benefit
High contrast ratio	High quality and control of S and P polarized light. better throughput	Superior Data Quality
Wide acceptance angle	Polarization of full spectrophotometer beam without compromise	Improving throughput leading to improved system sensitivity.
Very thin only 3 mm	Is smaller and more compact than Glan-Taylor/Thompson	extra room in the sample compartment to improve ease of analysis.
Wide wavelength range	250 nm – 2500 nm	No wavelength limitation during measurements

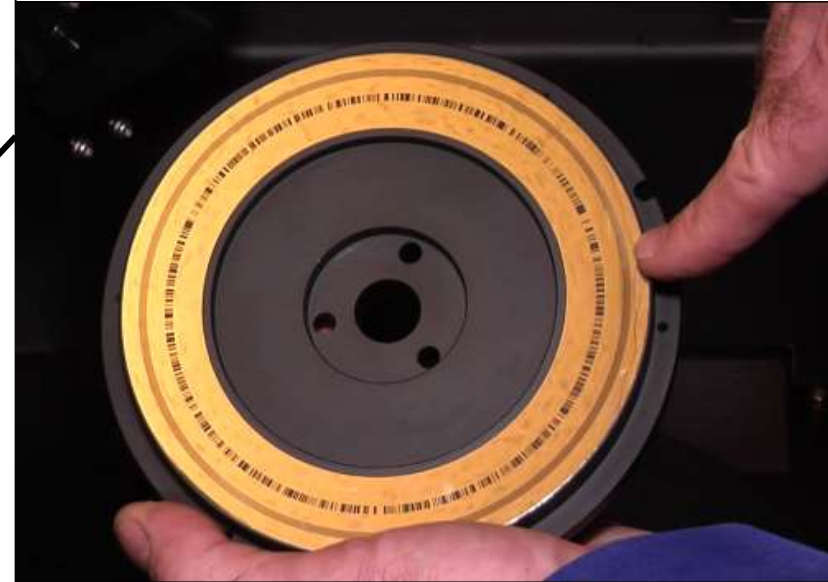




# Measure with 0.02° angular precision



## Unique High Resolution Optical Encoder



Feature	Advantage	Benefit
High Resolution Optical Encoder	Fine angular control of sample rotation and detector position to 0.02 deg.	Robustness/Reliability - system never misses a step and permits precise sample characterization



## P/N: G6874A, UMA Options

### Edge Mount Sample Holder

Adjustable Samples Sizes: From approx 1" (25.4 mm) to 6" (150 mm) in diameter.

Thicknness: Approx 200  $\mu\text{m}$  to 5 mm thick.

Non contact with front/back face of sample.

Max angular range is slightly limited due to "U" shaped frame around sample (sample diameter dependent).



Please note this picture is not anodized black yet.

## P/N: G6874A, UMA Options

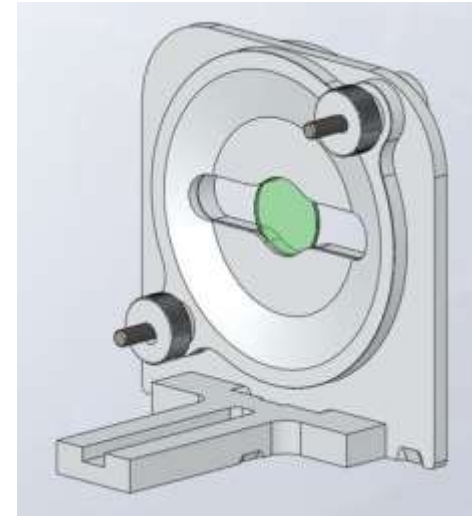
### Round Sample Holder

Sample Sizes:

1" and 2" round  
(2 mm selvage area)

Dedicated, simple, easy to use. Avoids contact with center of sample.

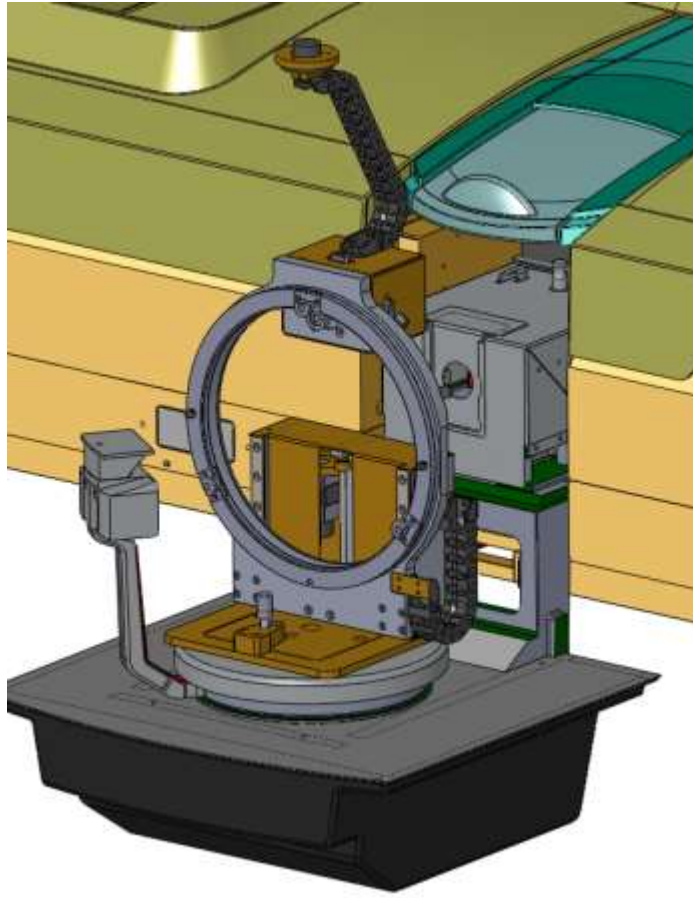
Ensure sample is not “offset” by a thick frame around optic, or that the frame increases the actual diameter of the sample. Samples sit in recessed rim of holder.



# Cube Beam Splitter Holder



# Solids Autosampler



**24/32 Multi Sample Holder**



**8" Wafer/Large Sample Holder**



Rotatable and translatable (x-phi) sample holder that mounts on the UMA rotational stage

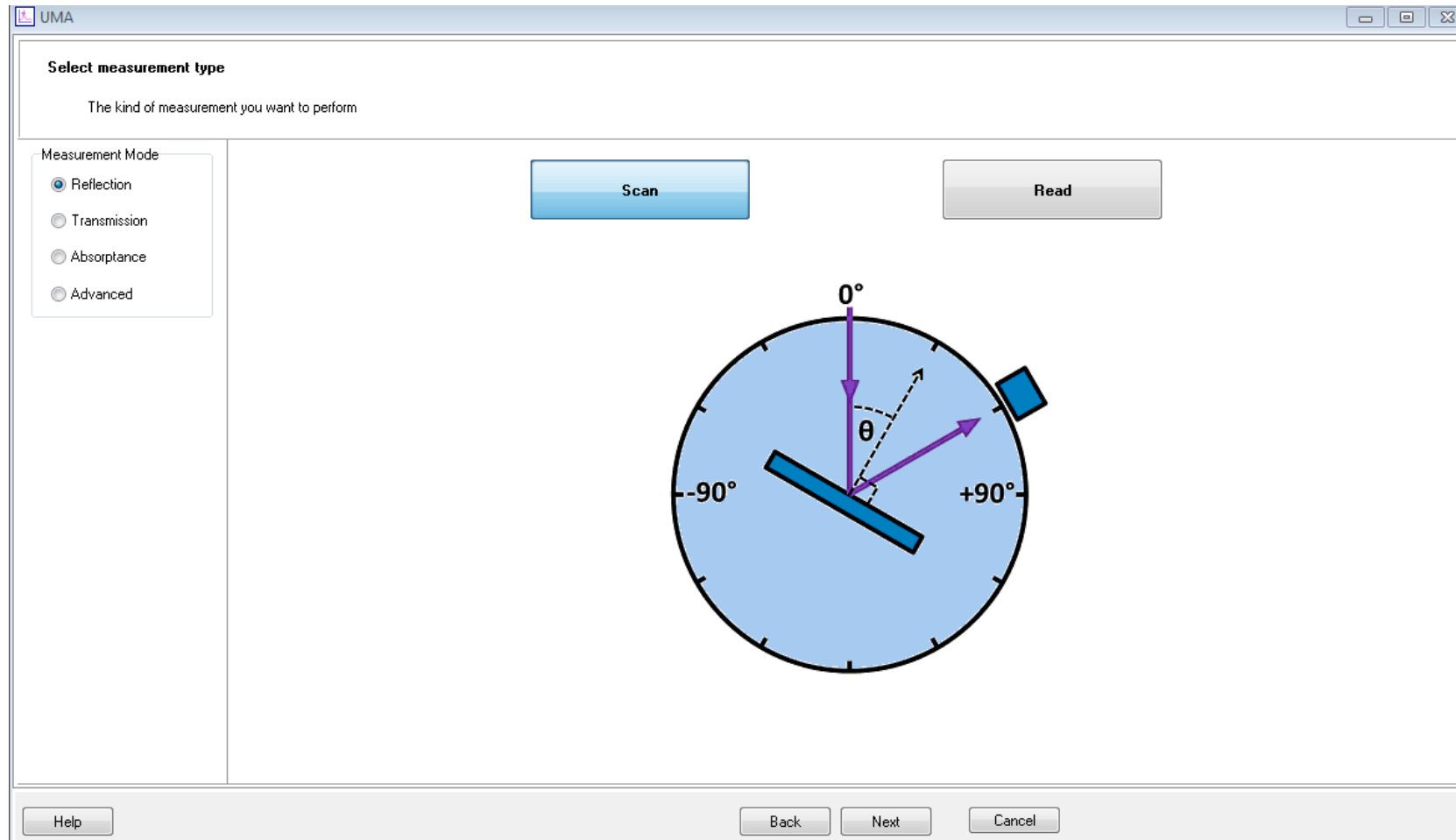
Allows rotation and translation of sample(s) for both T and R measurements at angle – multi-sample or “mapping”

Spatial resolution dictated by beam patch size at sample; 5x5 mm to 2x2 mm

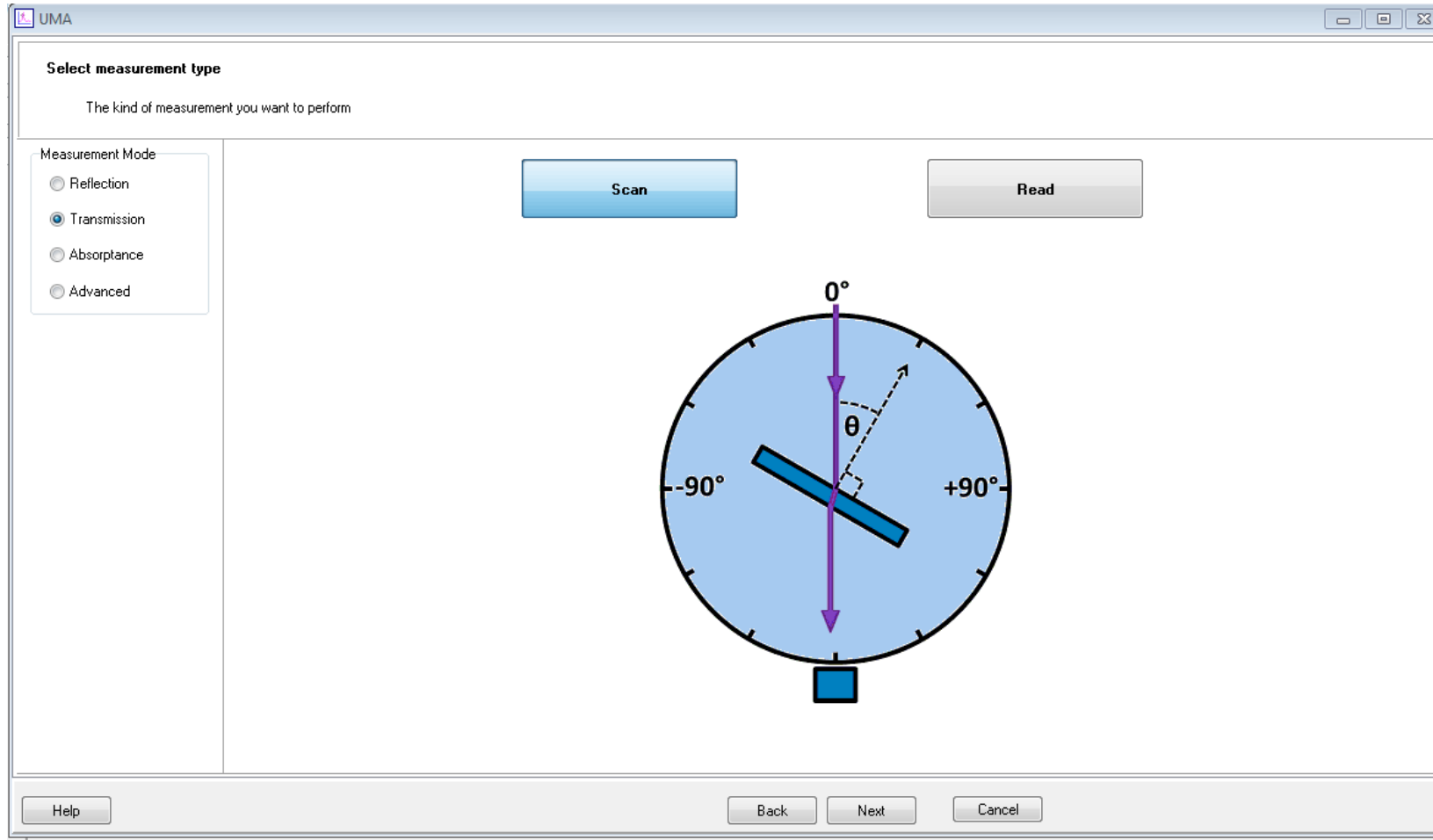
Potential 300% productivity increase!



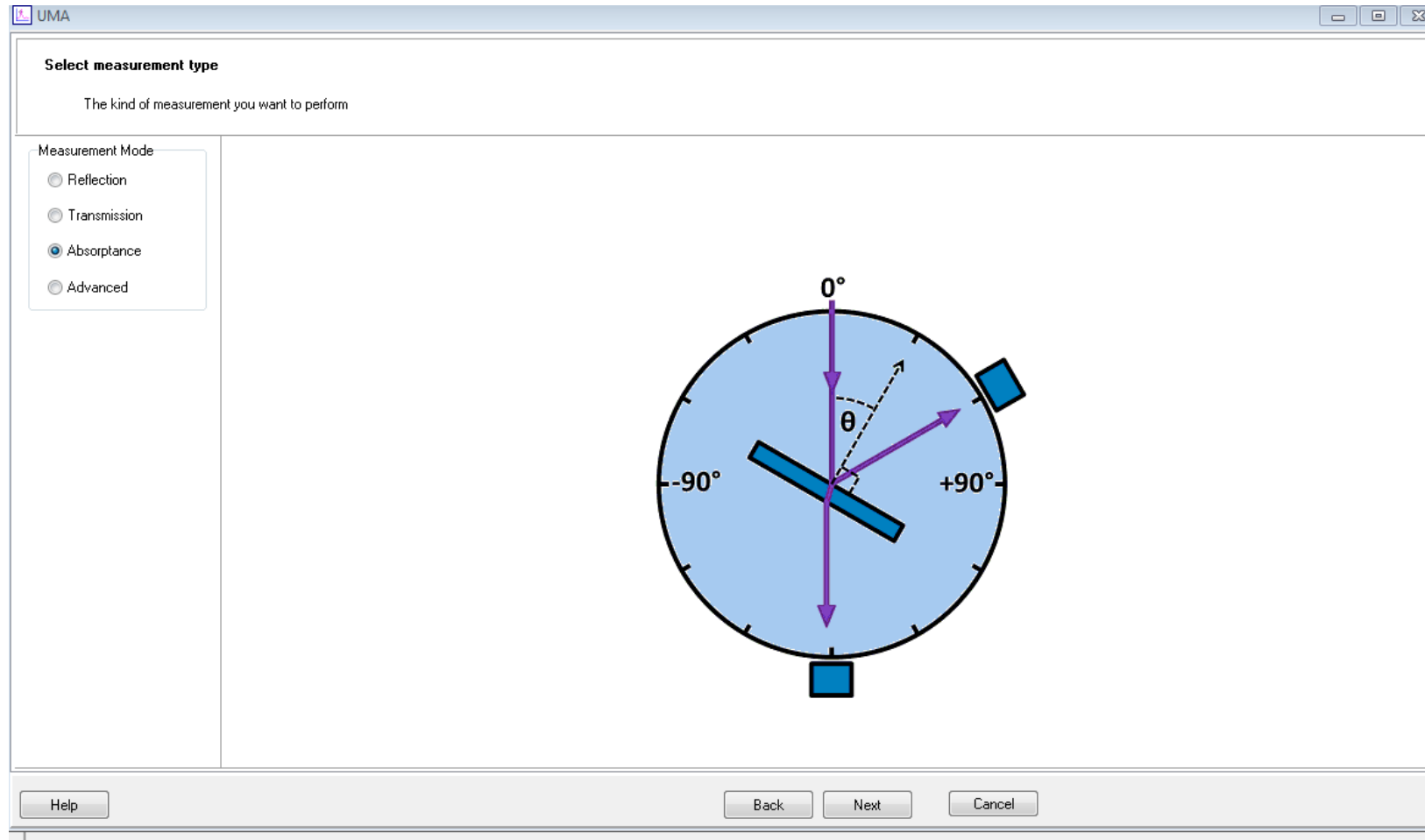
# Reflectance Measurement on UMA



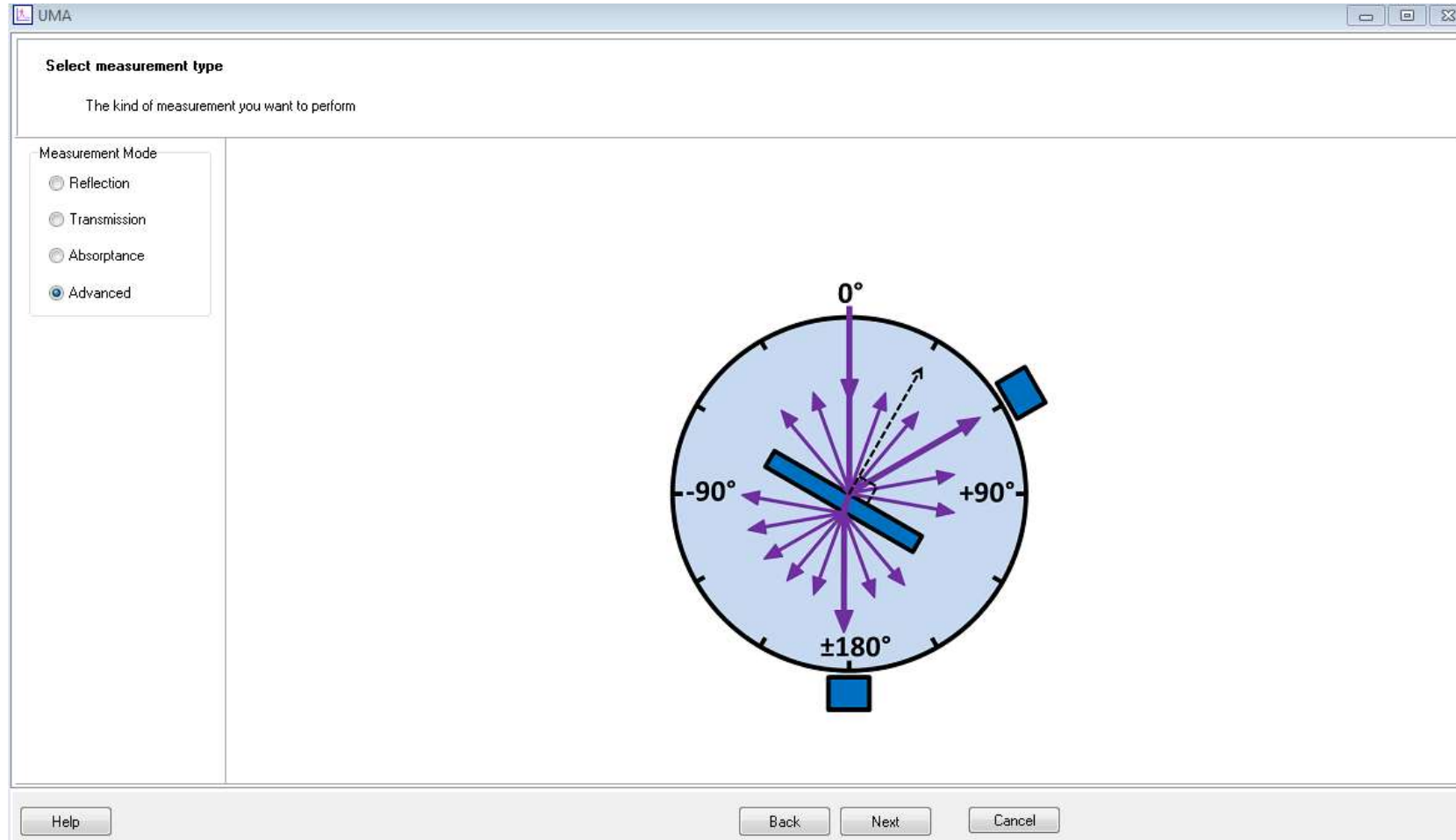
# Transmission Measurement on UMA



# Absorptance Measurement on UMA



# Advanced Measurement Capabilities

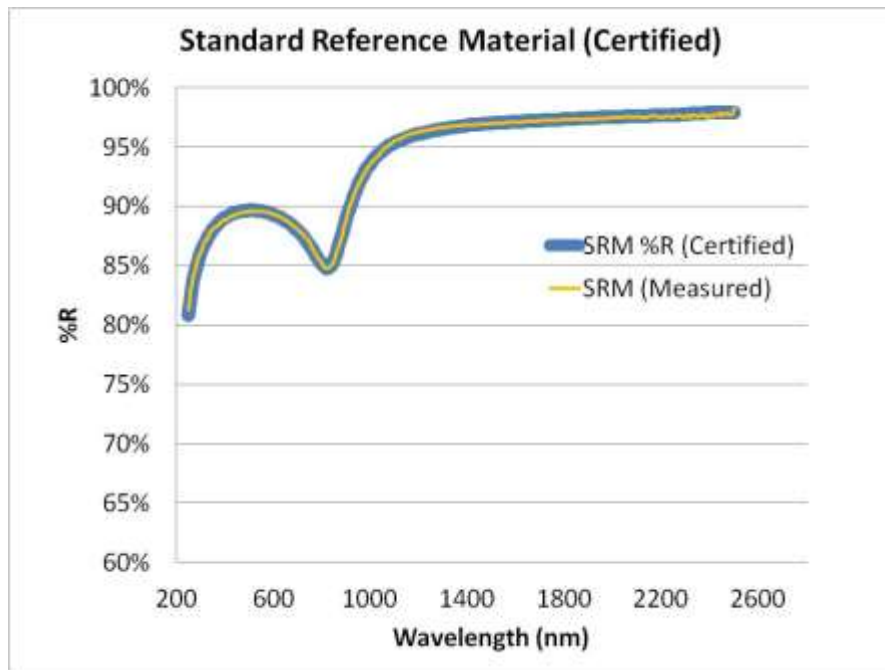


# Absolute Specular Reflection



## Summary

Measurement of absolute specular reflectance of a SRM traceable to a NIST standard.



## Results

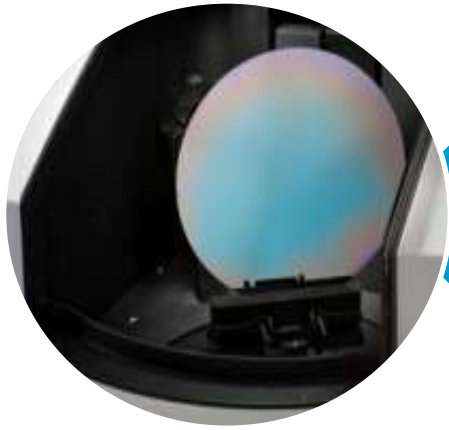
In this figure the measured and certified spectra have been overlaid

Comparisons between measured value and certified value can be seen to correspond very closely across the wavelength range 250 nm – 2500 nm. Data collect was collected in ~2 min scan.



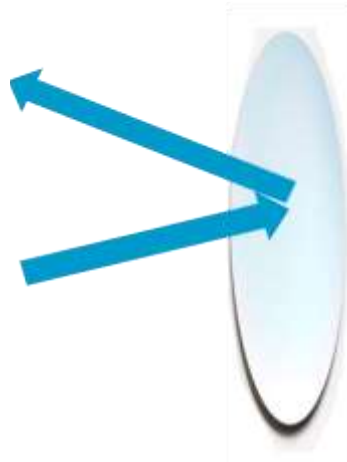


# Absolute Specular Reflection



## Application

Coating characterization and design validation. Comprehensive angular, and wavelength range, analysis of a coated silicon substrate, 200 mm diameter, 800  $\mu\text{m}$  thickness.

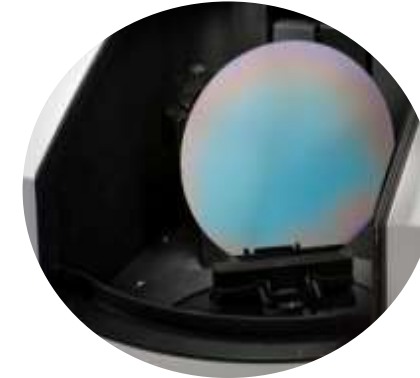
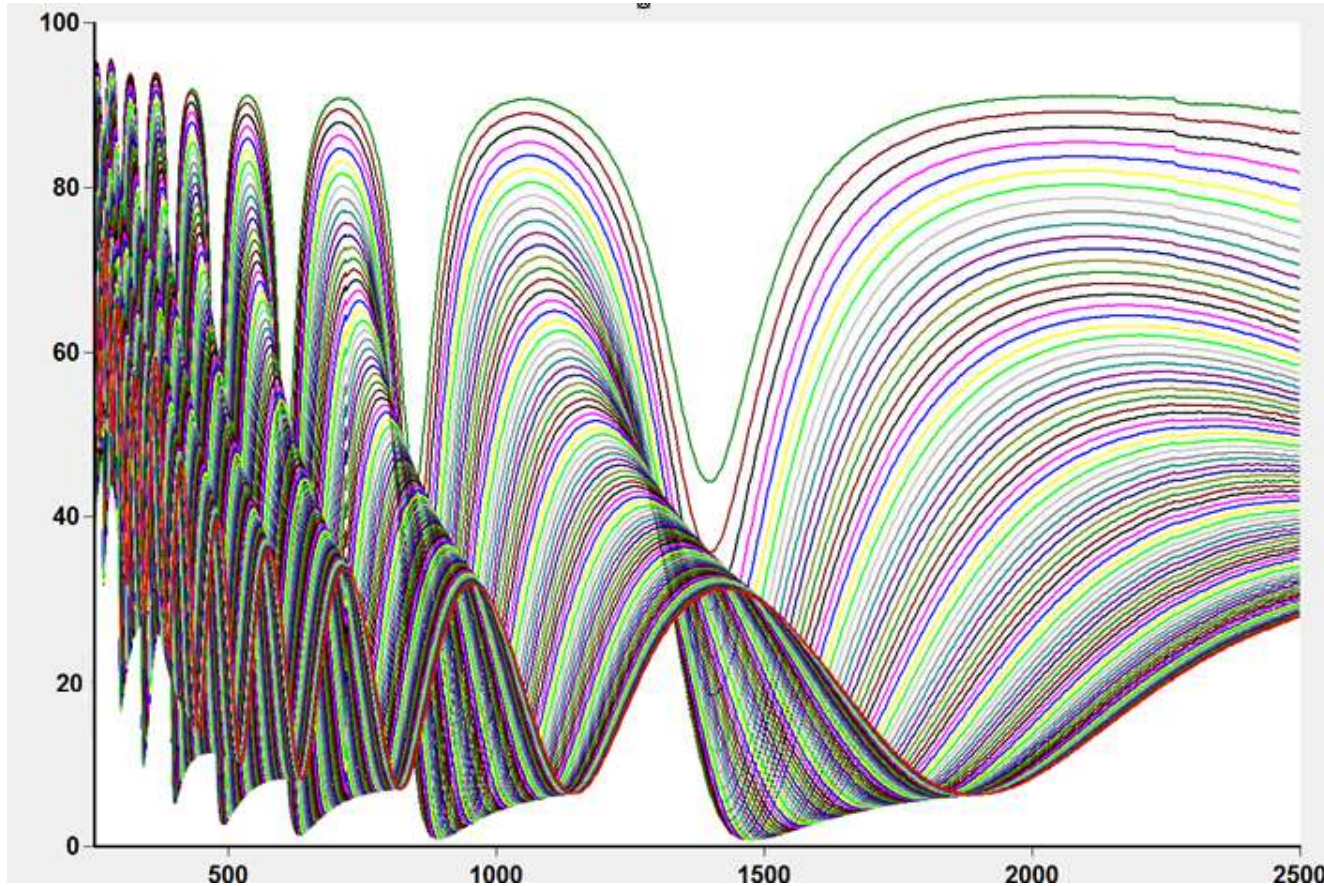


## Challenge

Efficient and accurate thin film design measurement by multi-angle, UV-Vis-NR spectroscopy and 2D contour plot visualization tools.



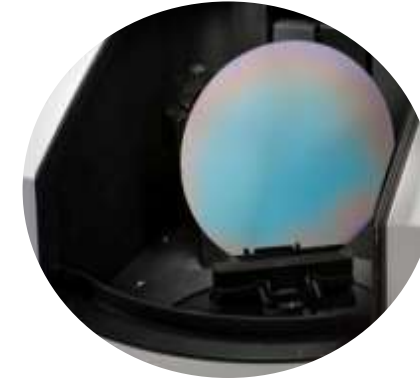
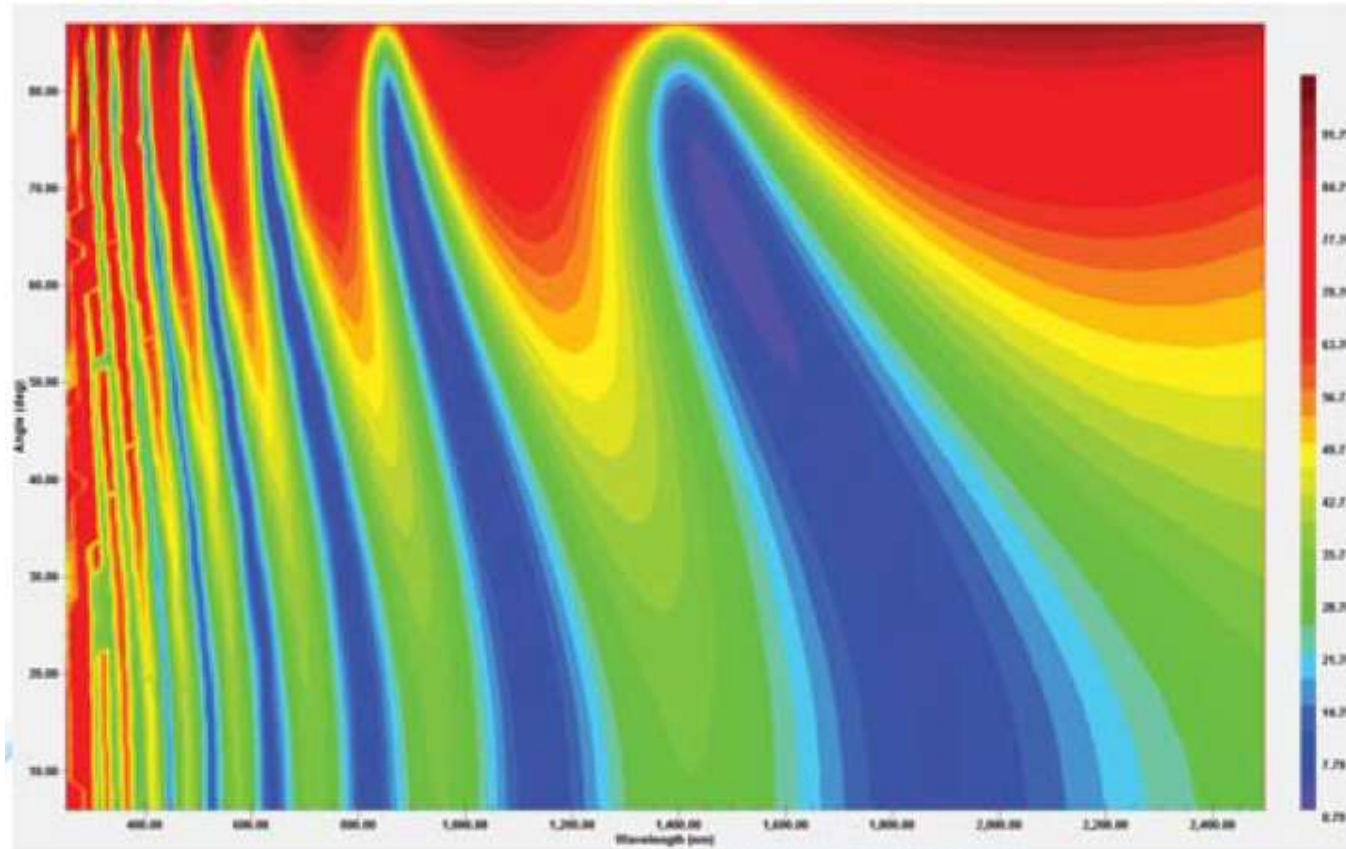
# Thin Film/Coatings, Specular Reflectance



## Results

Absolute specular reflectance measured of a coated silicon substrate in the UV-Vis-NIR from near normal angles of incidence (AOI) to high grazing angles. Spectra with AOI from 6 deg to 86 deg in 1 deg increments are shown for p-polarized light. The entire spectral collect was executed in a single unattended operation.

# Thin Film/Coatings, Specular Reflectance

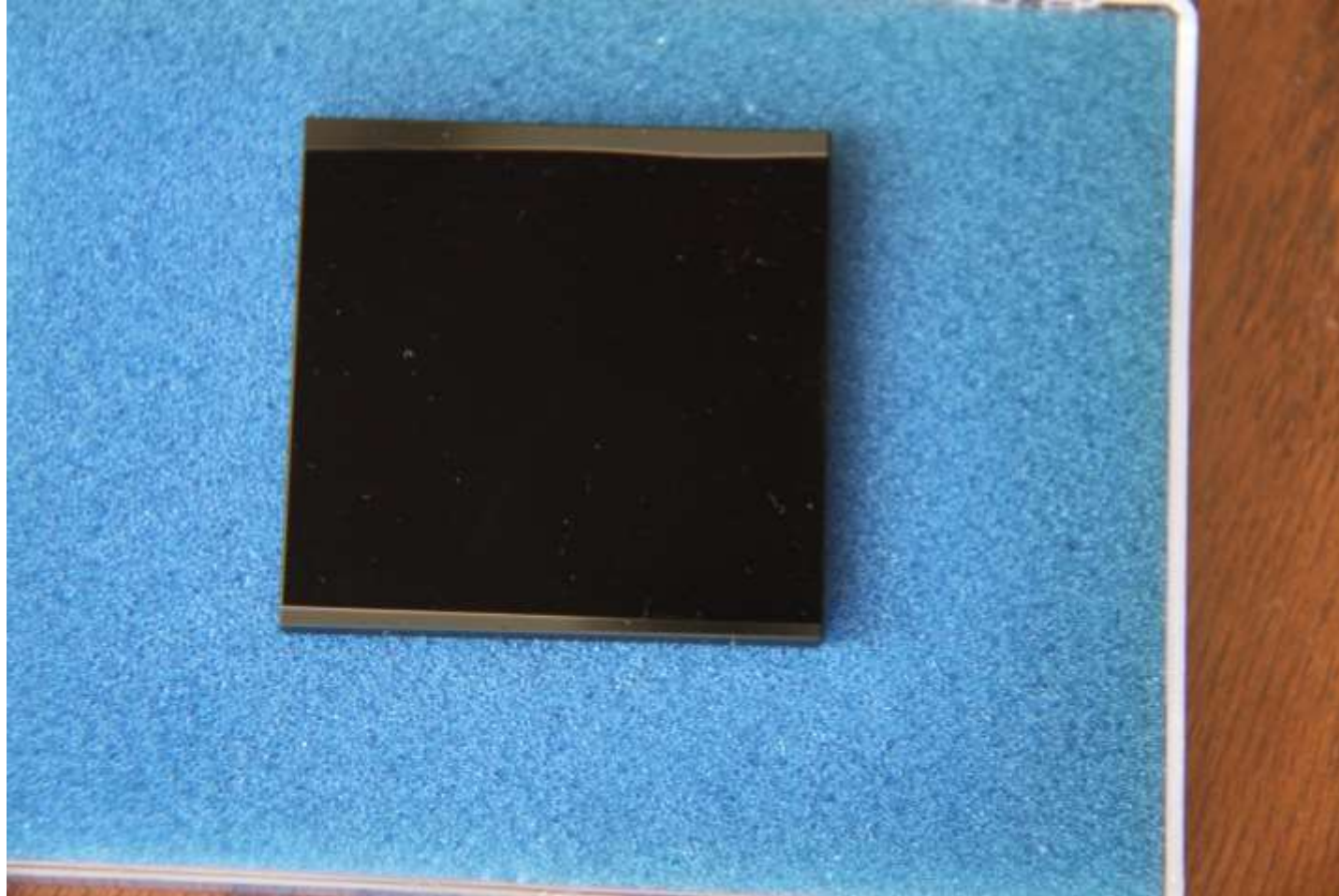


**Results**

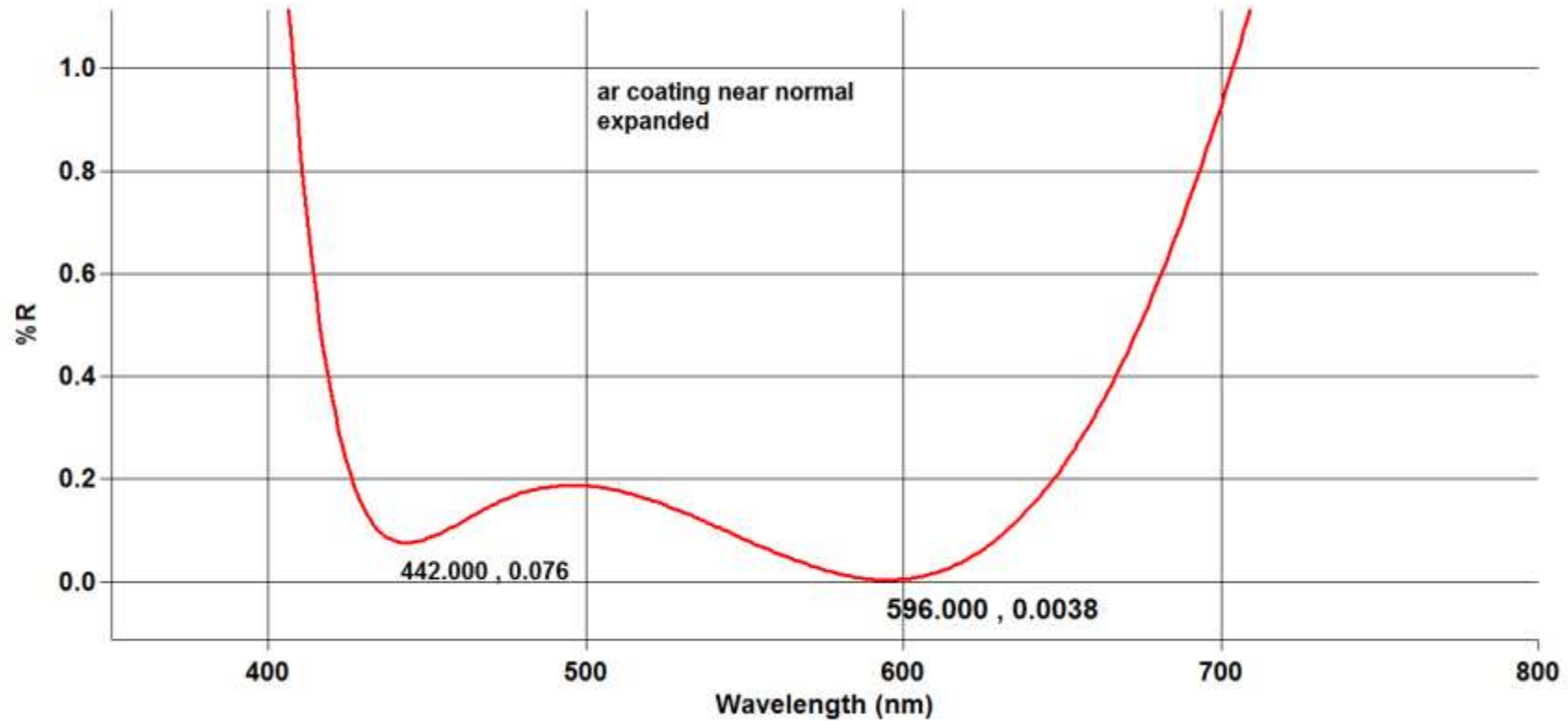
A 2D contour plot of the previous slide helps visualize the coating dependence with AOI and wavelength and aids with locating reflection minima and maxima, e.g., minimum reflection can easily identified at 1500 nm with 70 deg AOI.



# Example of ar Sample



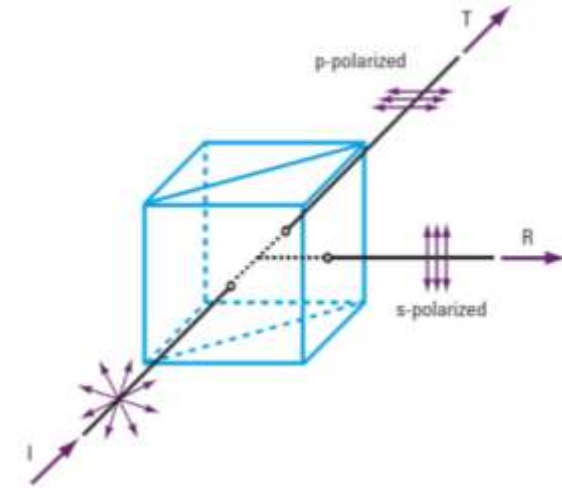
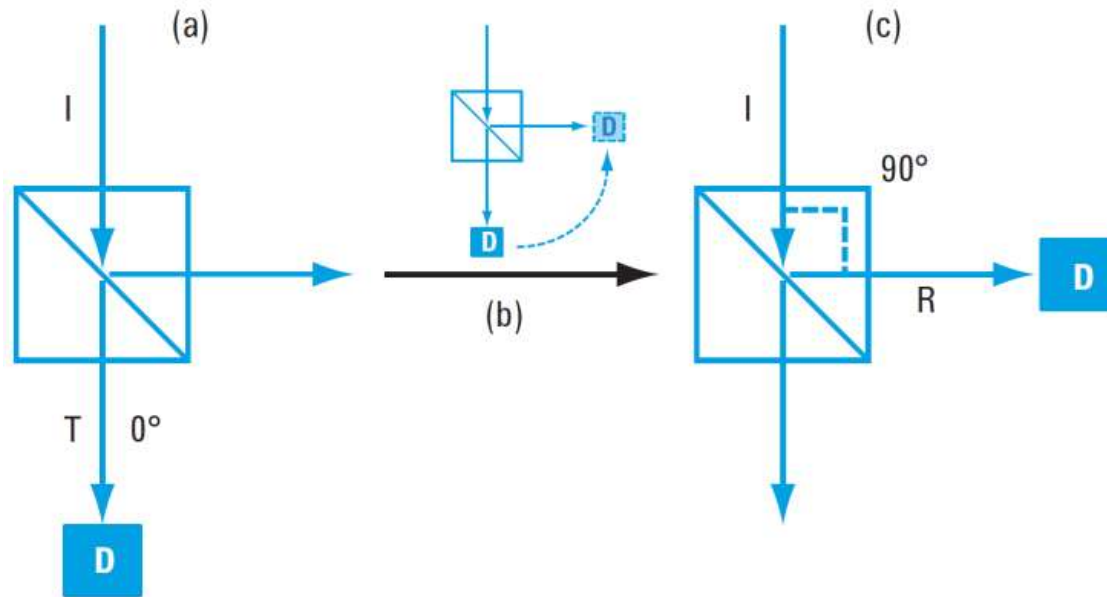
# ar Coating Near Normal Expanded



# Optics – Cube Beamsplitters

## Application Example

Polarizing CBS use in interferometer of nano-positioning system. Designed for HeNe laser @ 632.8 nm. Proprietary beamsplitter & anti-reflection coating made from  $\text{TiO}_2$  &  $\text{SiO}_2$ . The two prisms are bonded with optical adhesive.



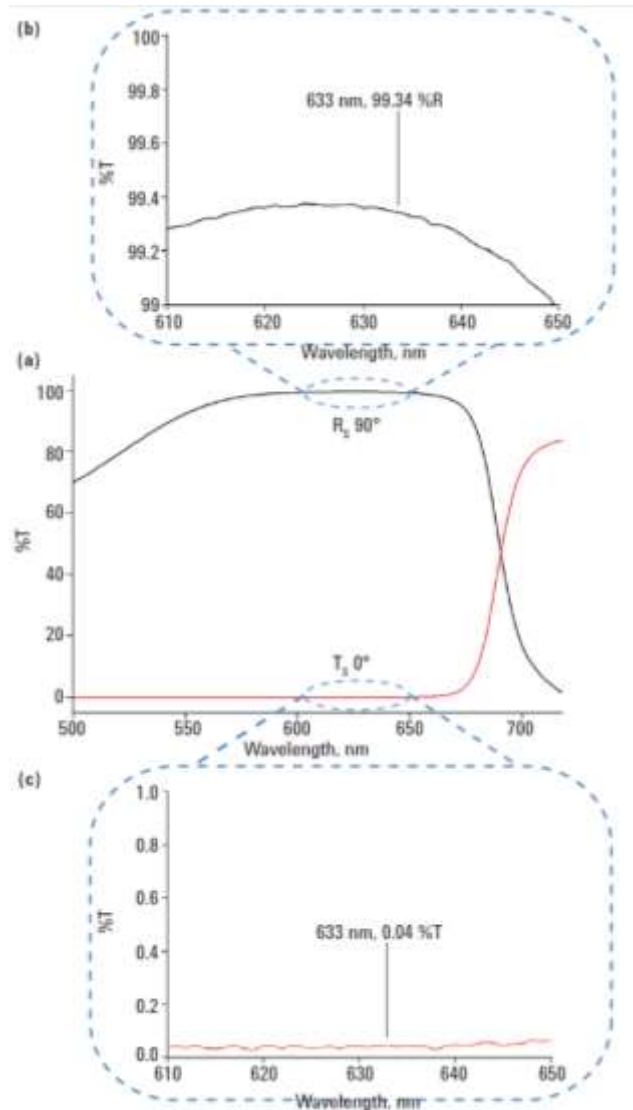
## The Challenge

Measure  $T$  and  $R$  for s- and p-polarized incident without moving sample and therefore light incident on the sample. On the UMS this is easy.... AND FAST!





# Optics – Cube Beamsplitters

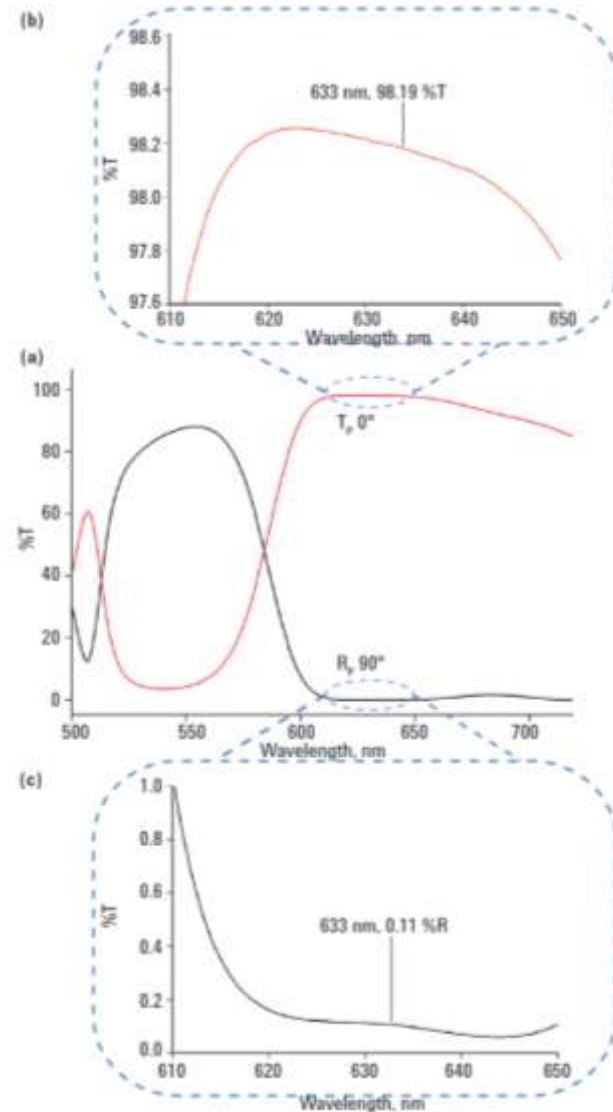


## Results

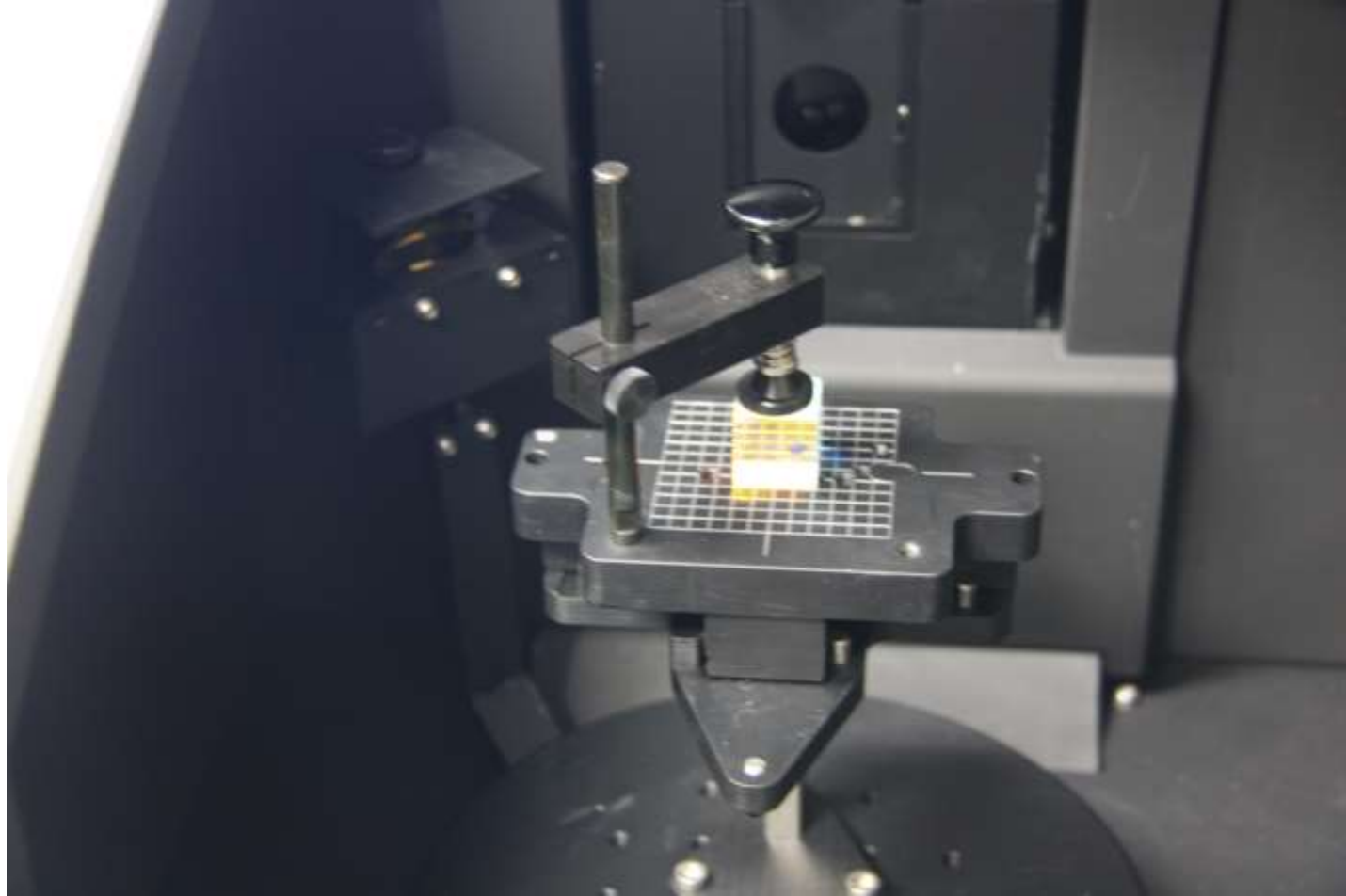
S-polarized spectra (left) desire High  $R_s$  and Low  $T_s$ .

P-polarized spectra (right) desire High  $T_p$  and Low  $R_p$ .

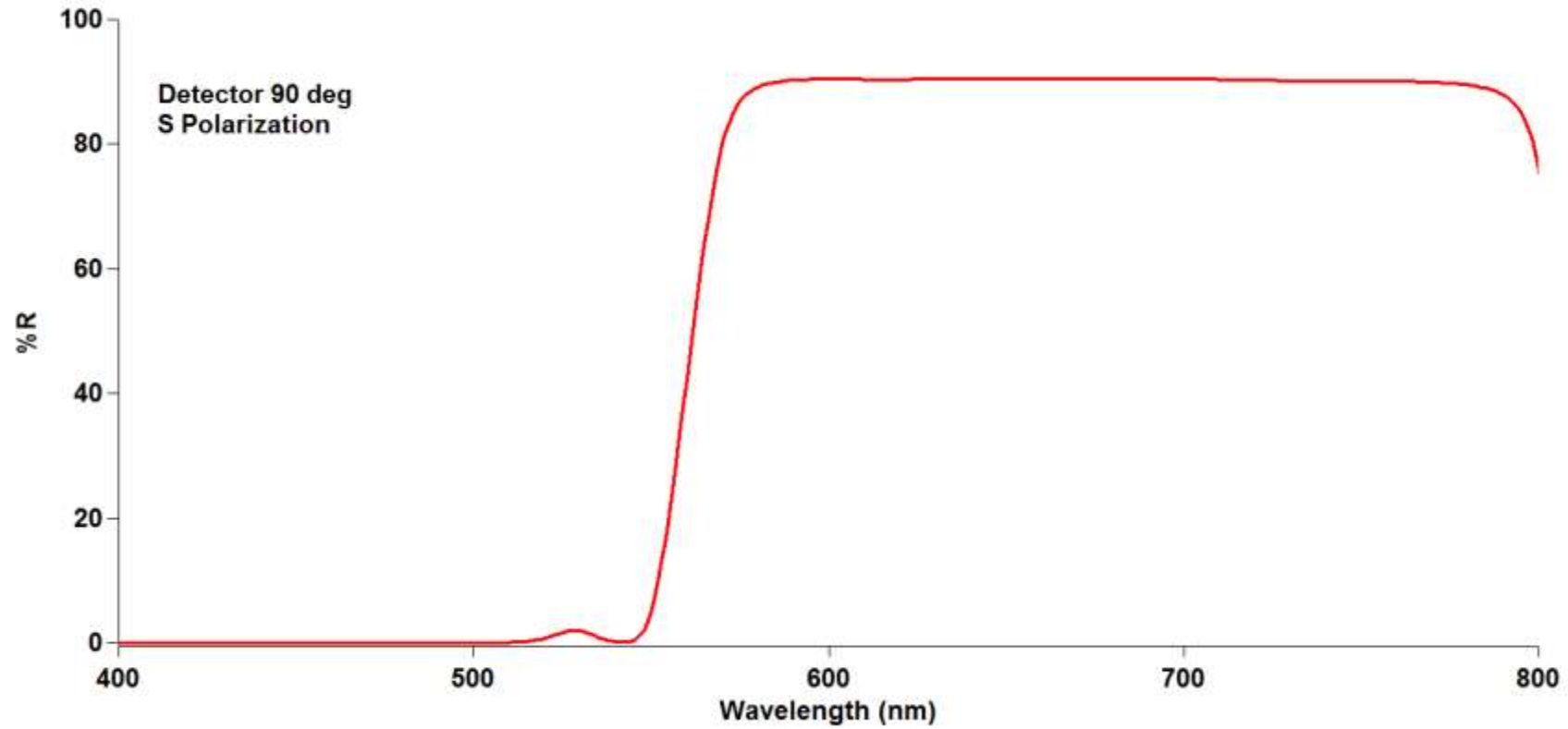
Performance is easily determined. Results can be fed back into CBS design.



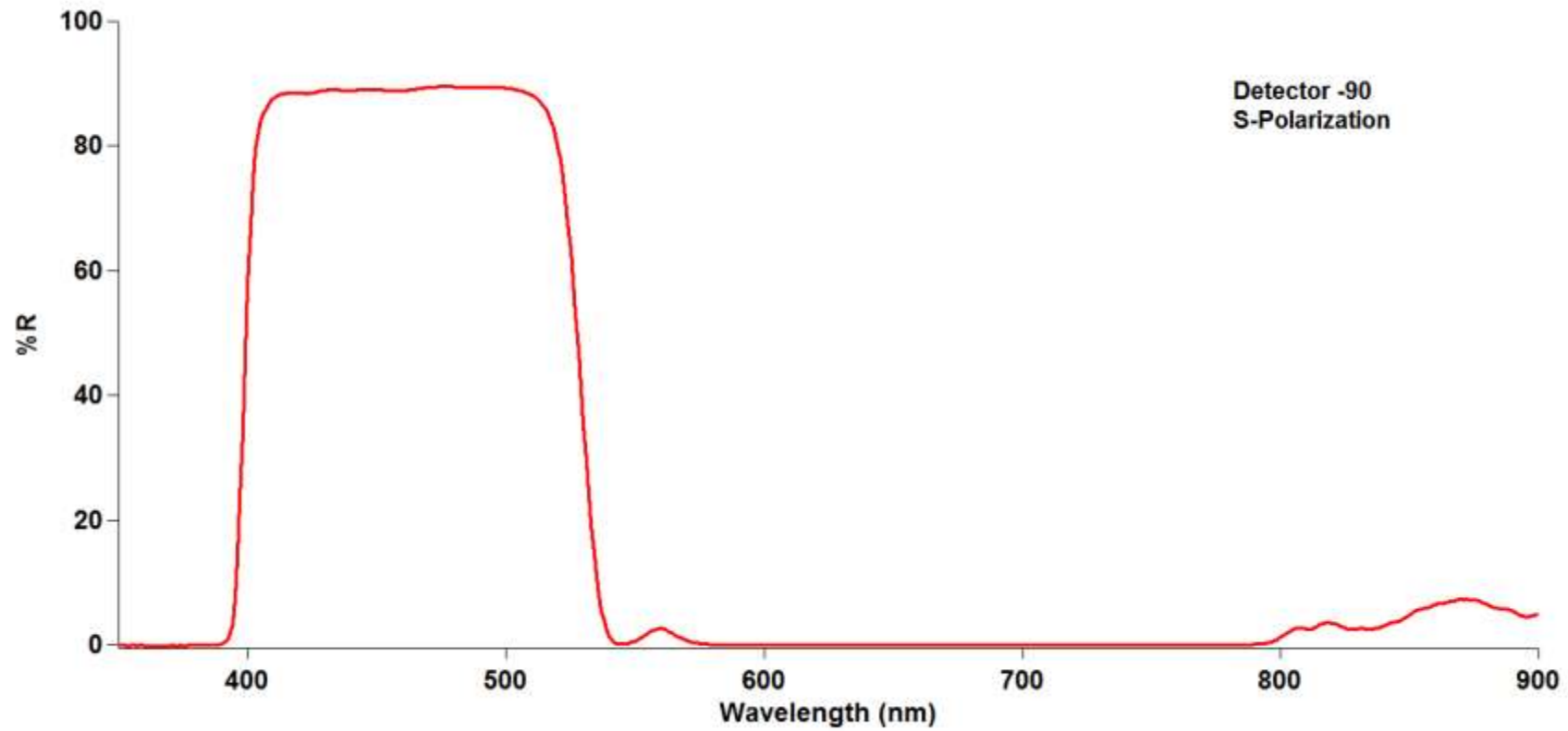
# RGB Cube Prism



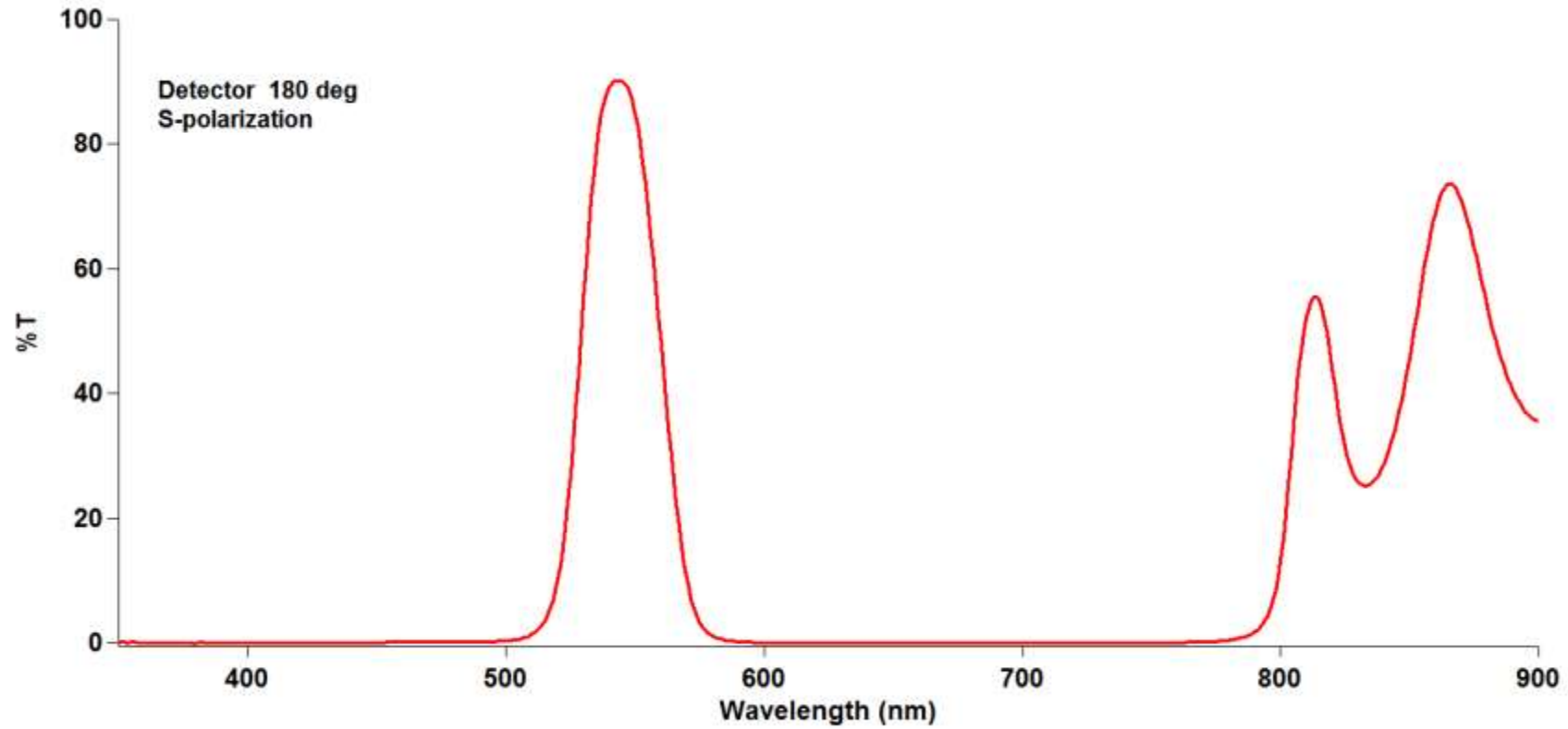
# RGB Cube Prism



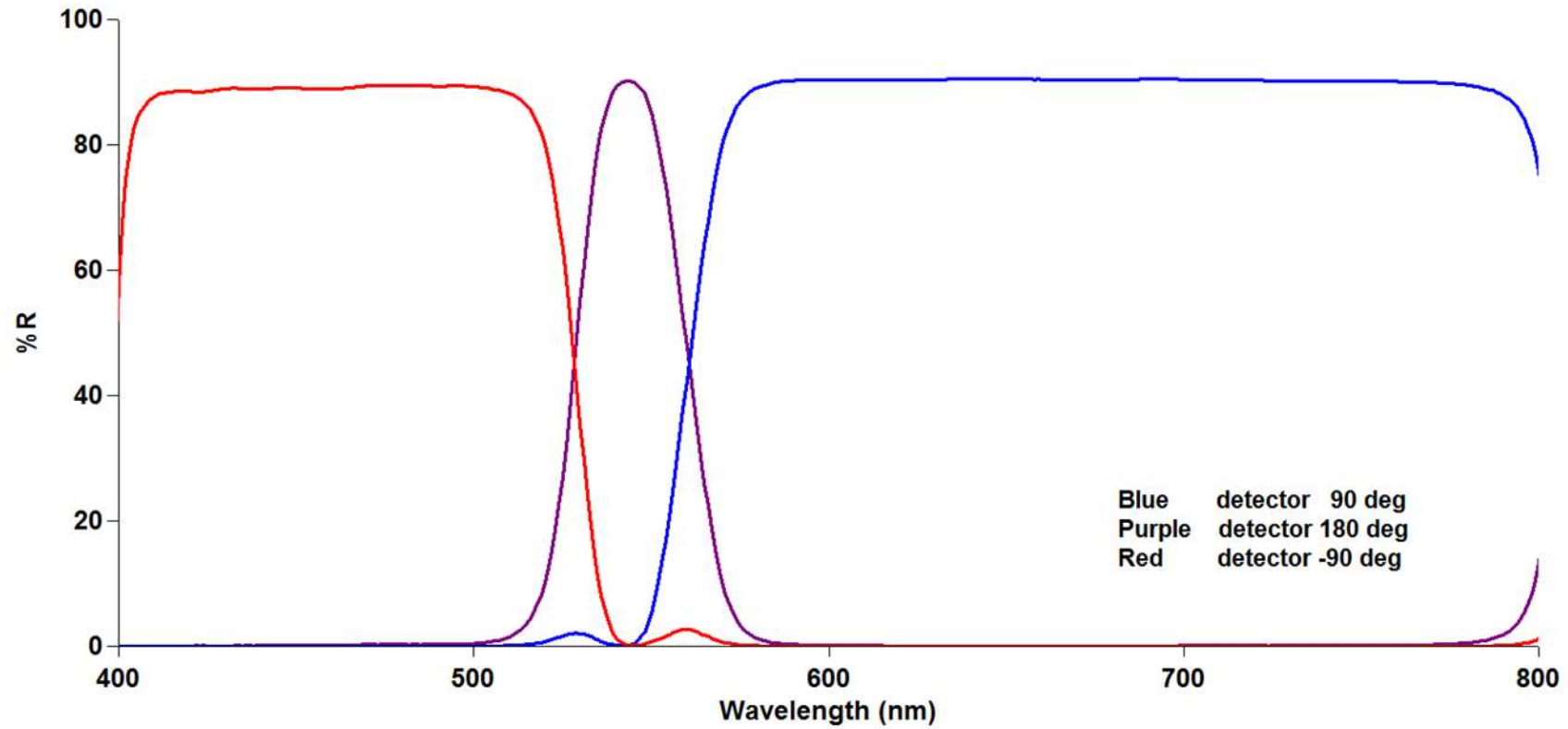
# RGB Cube Prism



# RGB Cube Prism

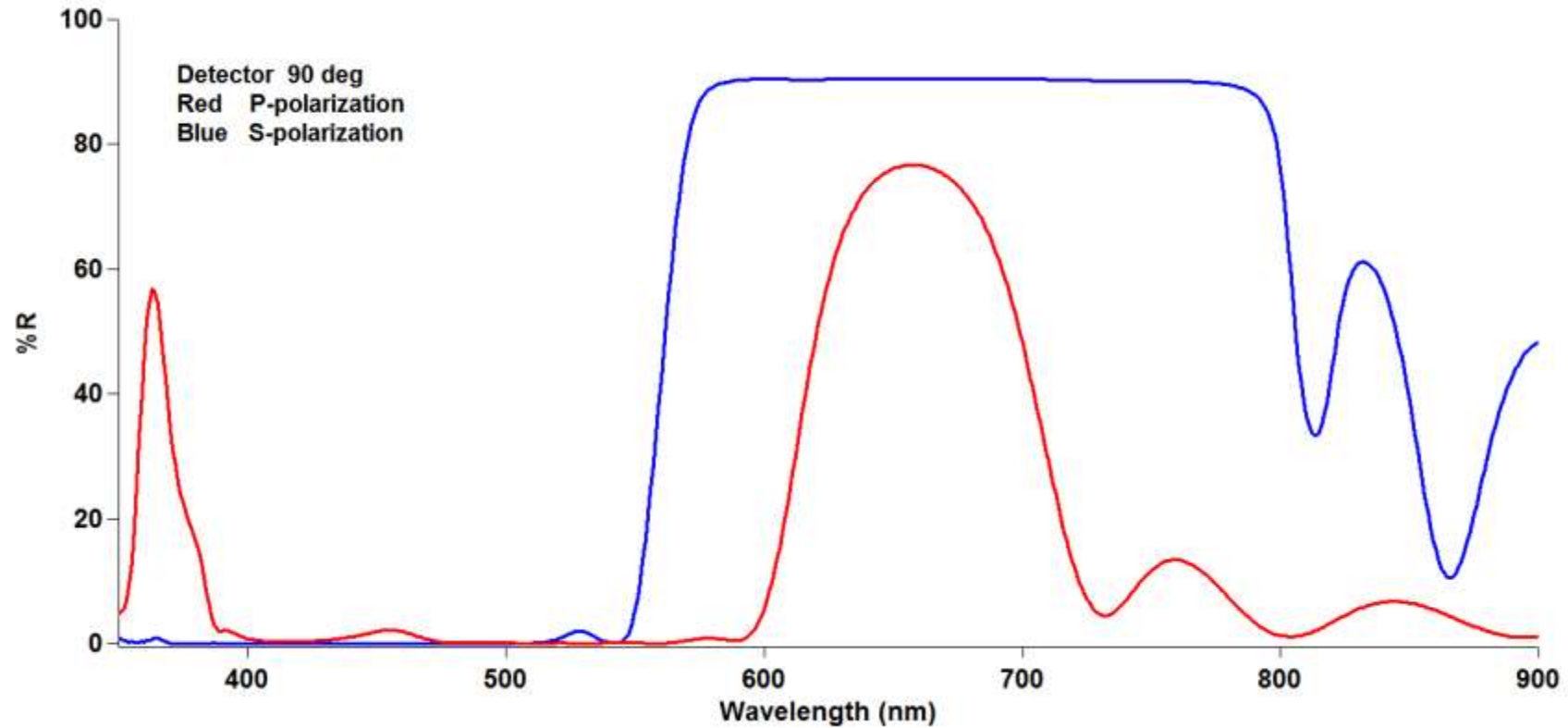


# RGB Cube Prism Overlay of Data



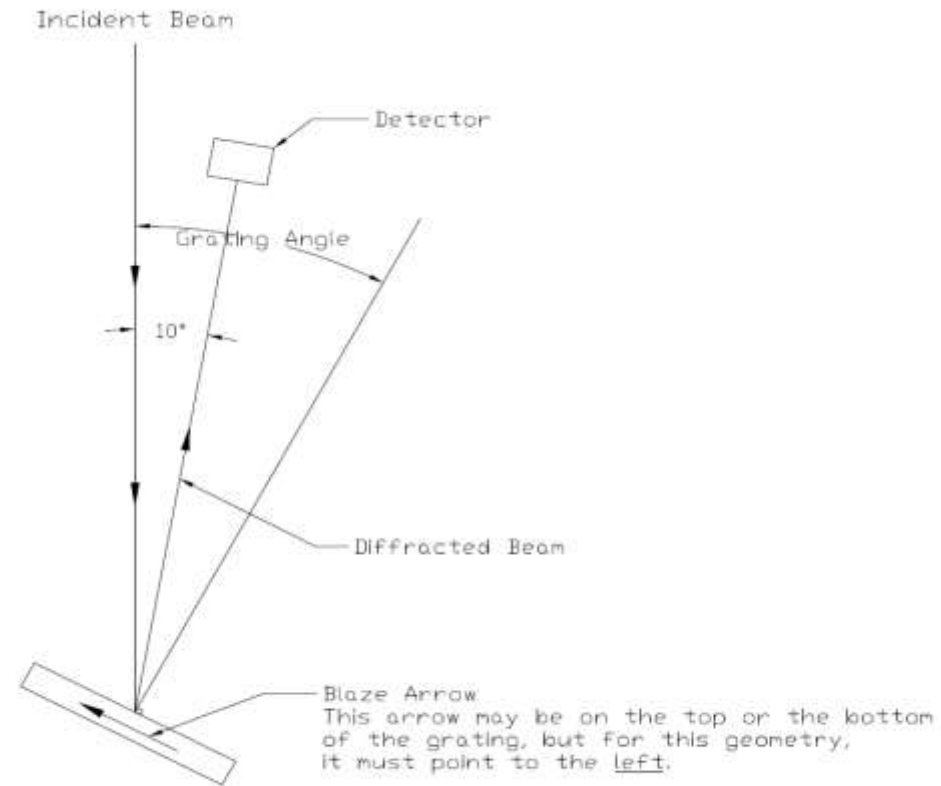


# RGB Cube Prism Effect of Polarization



# Measuring Grating Efficiency

# Orientation of Sample



# GRATING RESULTS

Samples Run :

300 lines per mm

1200 lines per mm

1800 lines per mm

3600 lines per mm

Data Collected at a Data Interval of 1 nm and 10 nm

Data Collected with SBW of 2 nm and 5 nm

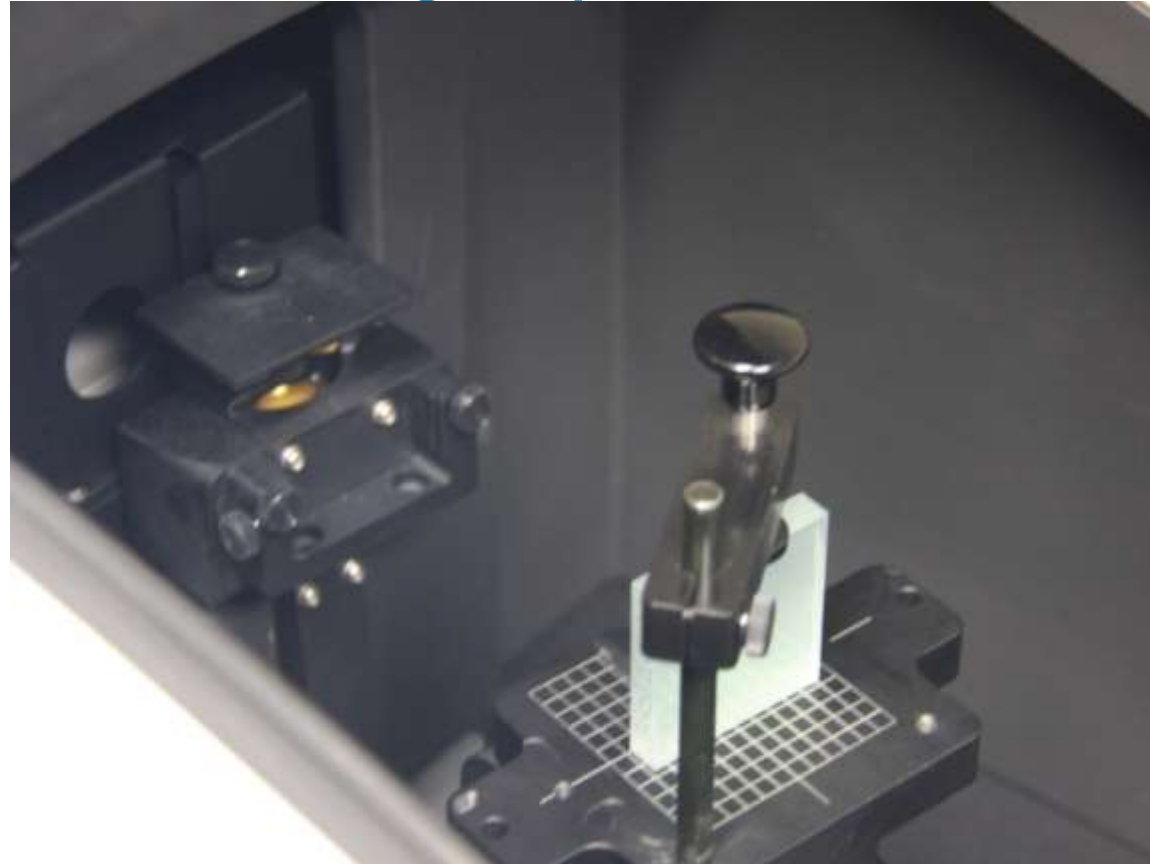


## Grating Sample on Cube Sample Holder



**300 line per mm Grating on Sample Holder**

## Grating Sample in UMA



**Detector Held at 10 degrees**

**Sample Stage Rotated to Calculated Angles for Grating and Wavelength which have been read from a csv file**

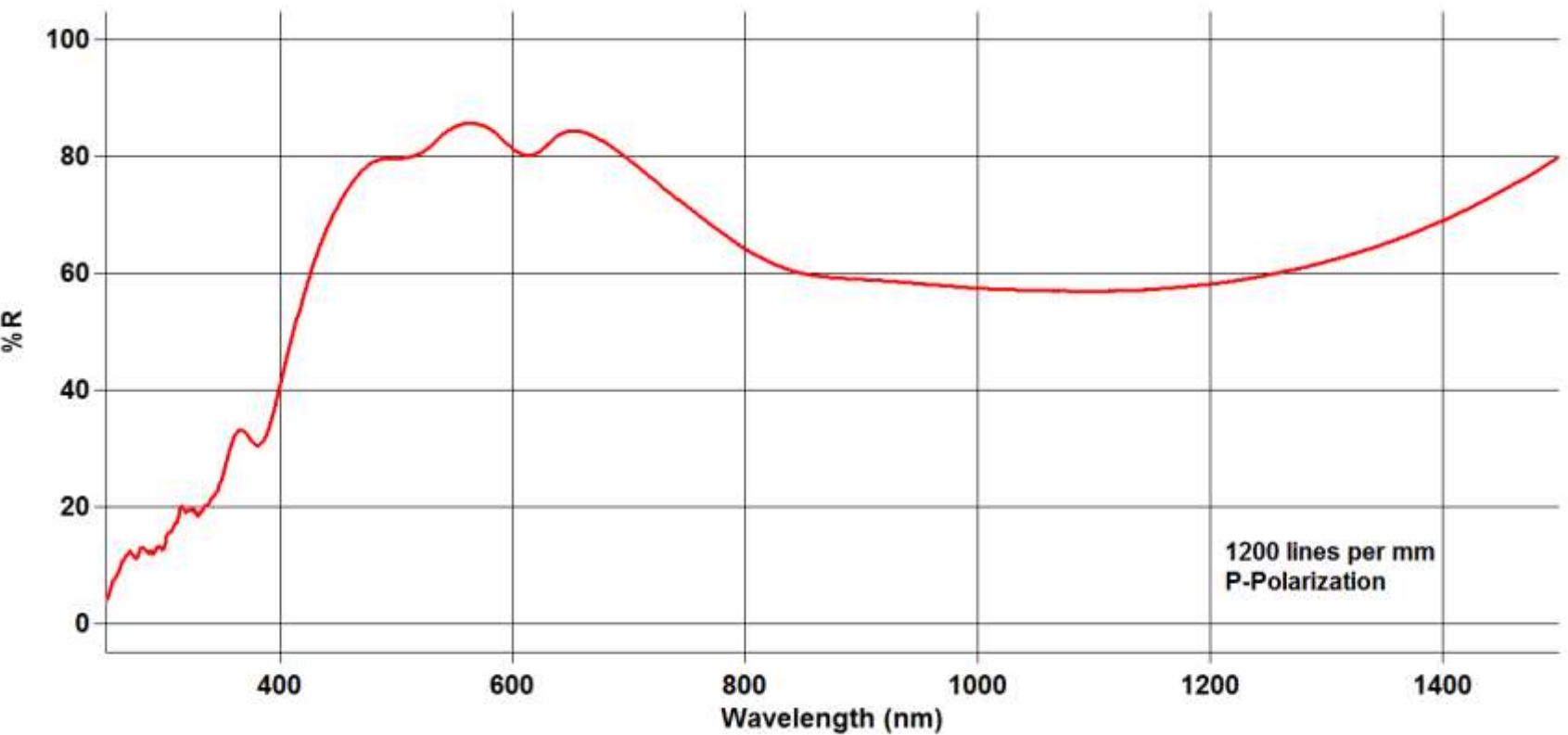




**1200 lines per mm Sample**  
**Data Interval 1 nm SBW 2 nm**



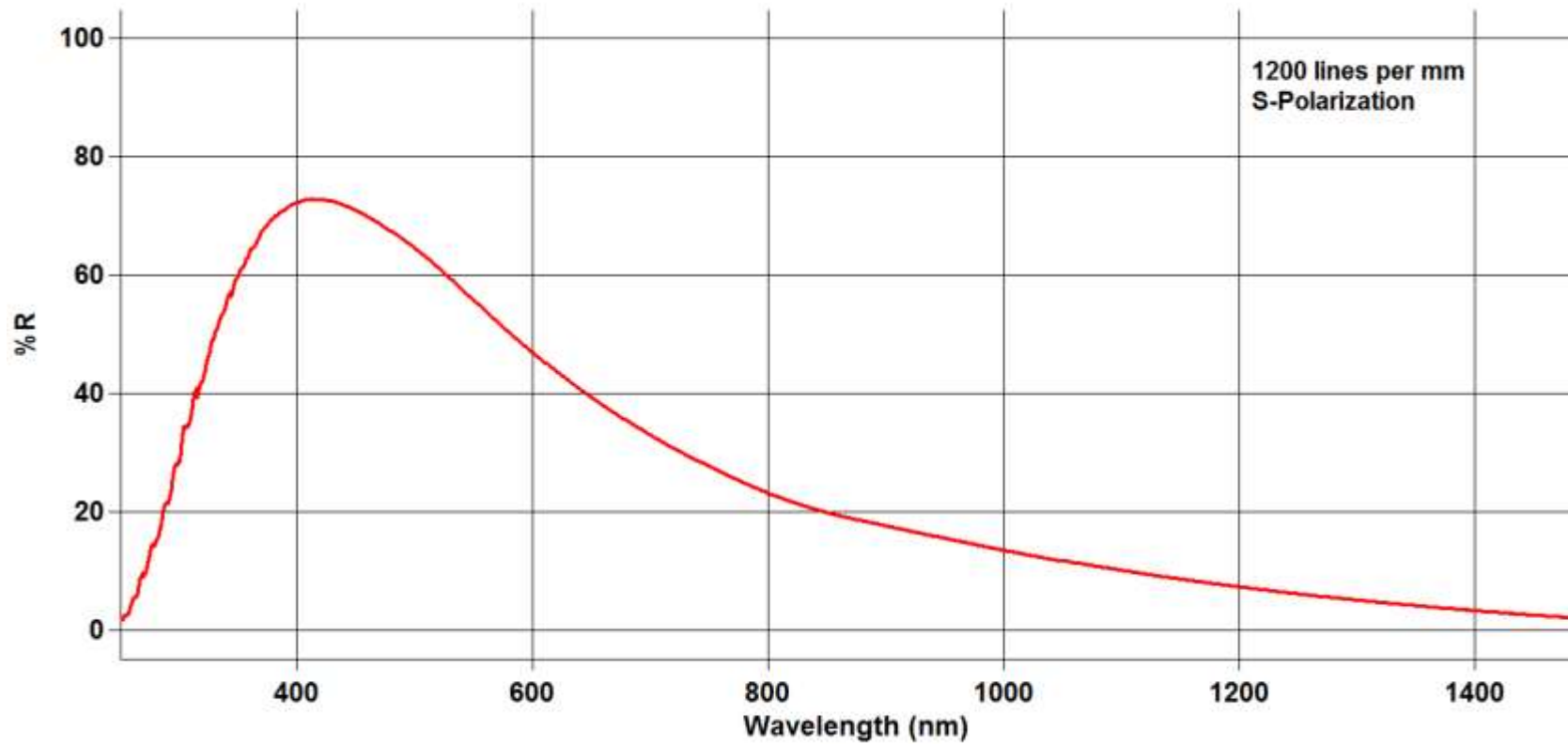
# 1200 lines per mm Grating



Data Interval 1 nm SBW 2 nm

P-Polarization

## 1200 lines per mm Grating



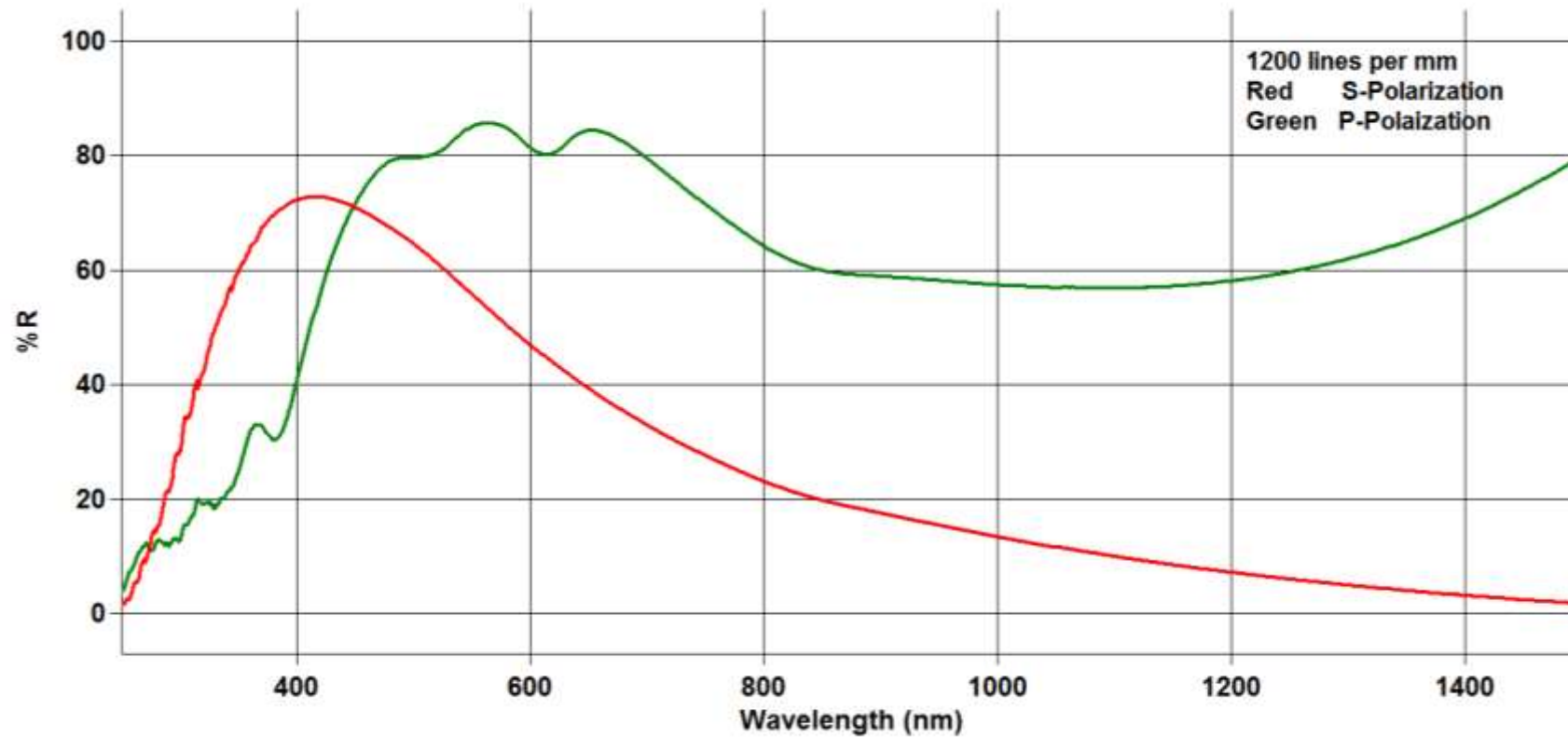
Data Interval 1 nm SBW 2 nm

S-Polarization



Agilent Technologies

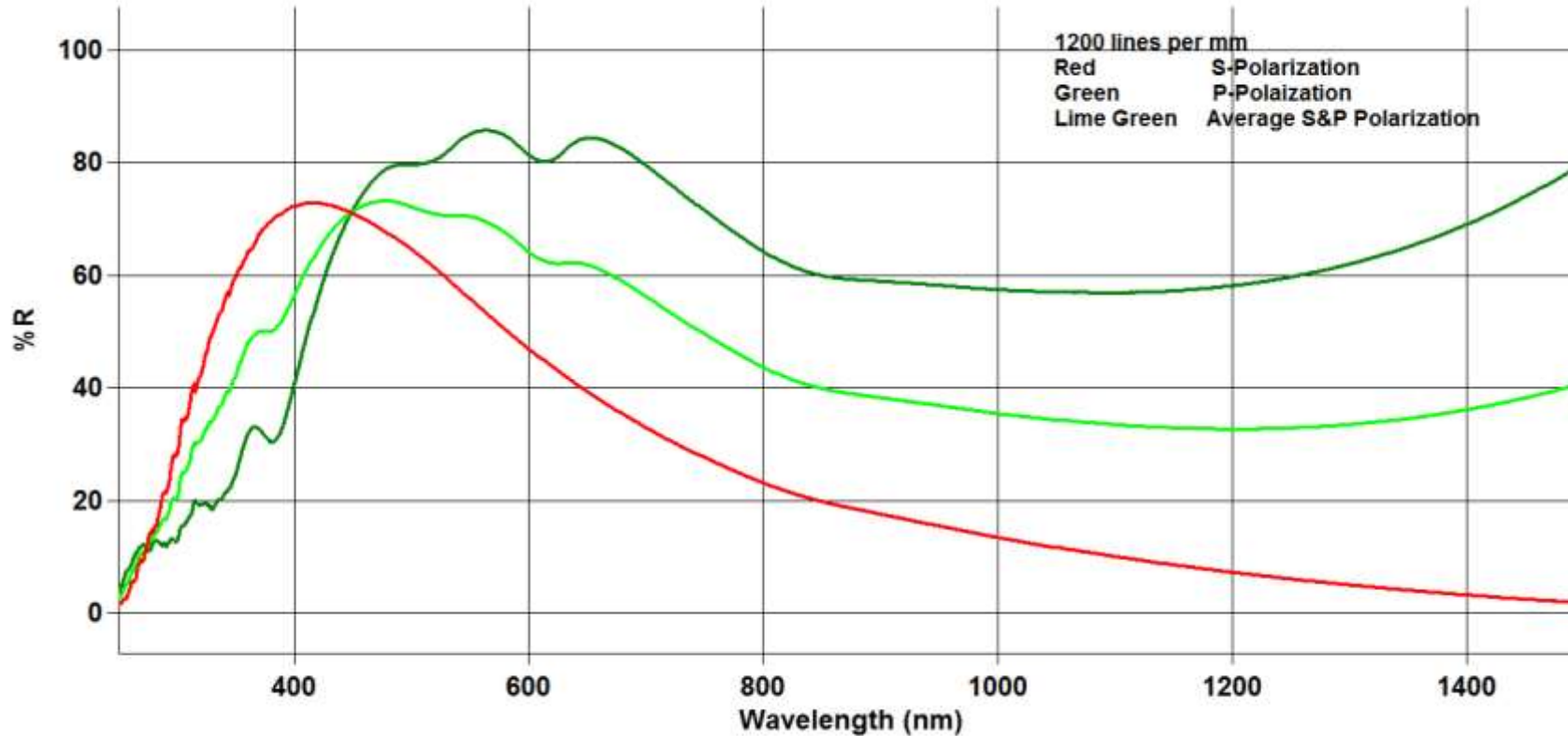
## 1200 lines per mm Grating



Overlay of S&P Polarization

Data Interval 1 nm SBW 2 nm

## 1200 lines per mm Grating



Overlay of S, P, and Average Polarization

Data Interval 1 nm SBW 2 nm

## Elapsed Time for Data Collection for 1200 lines per mm Sample

1200 lines per mm P-Polarization

Start Time 5:46:57 PM

End Time 7:56:59 PM

Elapsed Time = 2 hrs 10 min 02 sec

1200 lines per mm S-Polarization

Start Time 10:44:27 PM

End Time 12:55:53 AM

Elapsed Time = 2 hrs 11 min 28 sec

Total Elapsed Time = 4 hrs 21 min 30 sec

Number of data points per scan = 1251



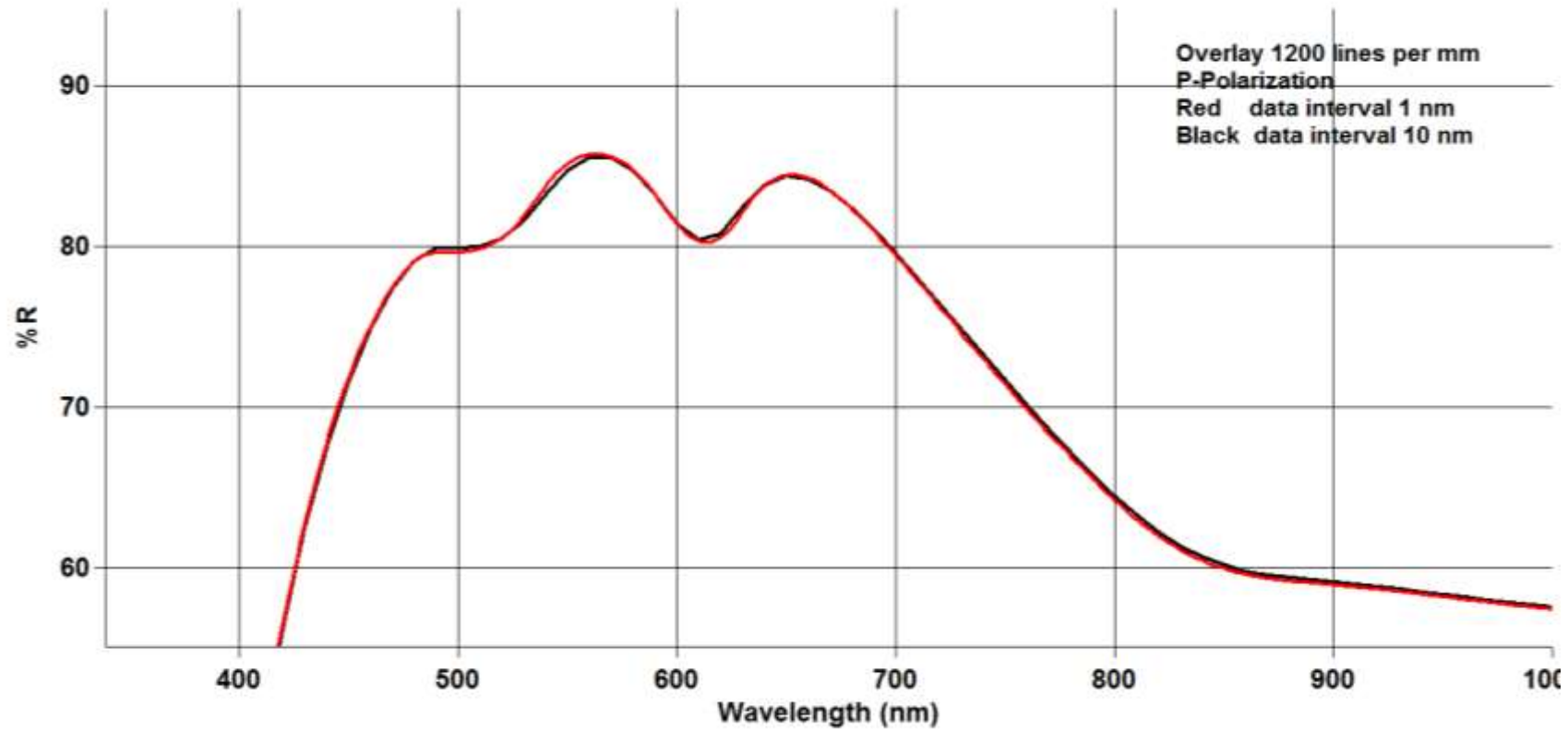
# Comparison of Data Interval of 1 nm and 10 nm

## Sample 1200 lines per mm





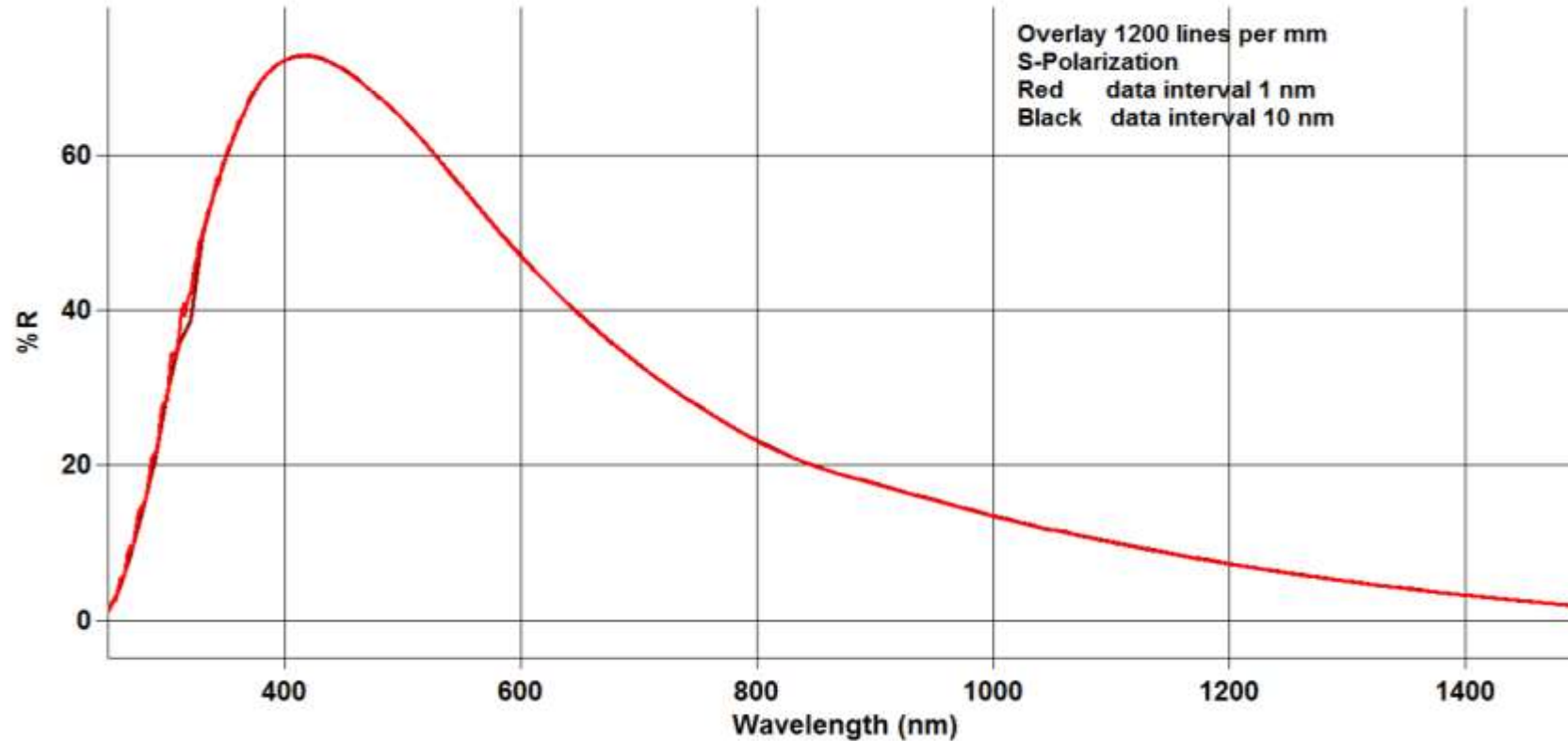
## 1200 lines per mm Grating



Overlay of P-Polarization Data Interval of 1 nm and 10 nm

Red data interval = 1 nm Black data interval = 10 nm

## 1200 lines per mm Grating



Overlay of S-Polarization Data Interval of 1 nm and 10 nm

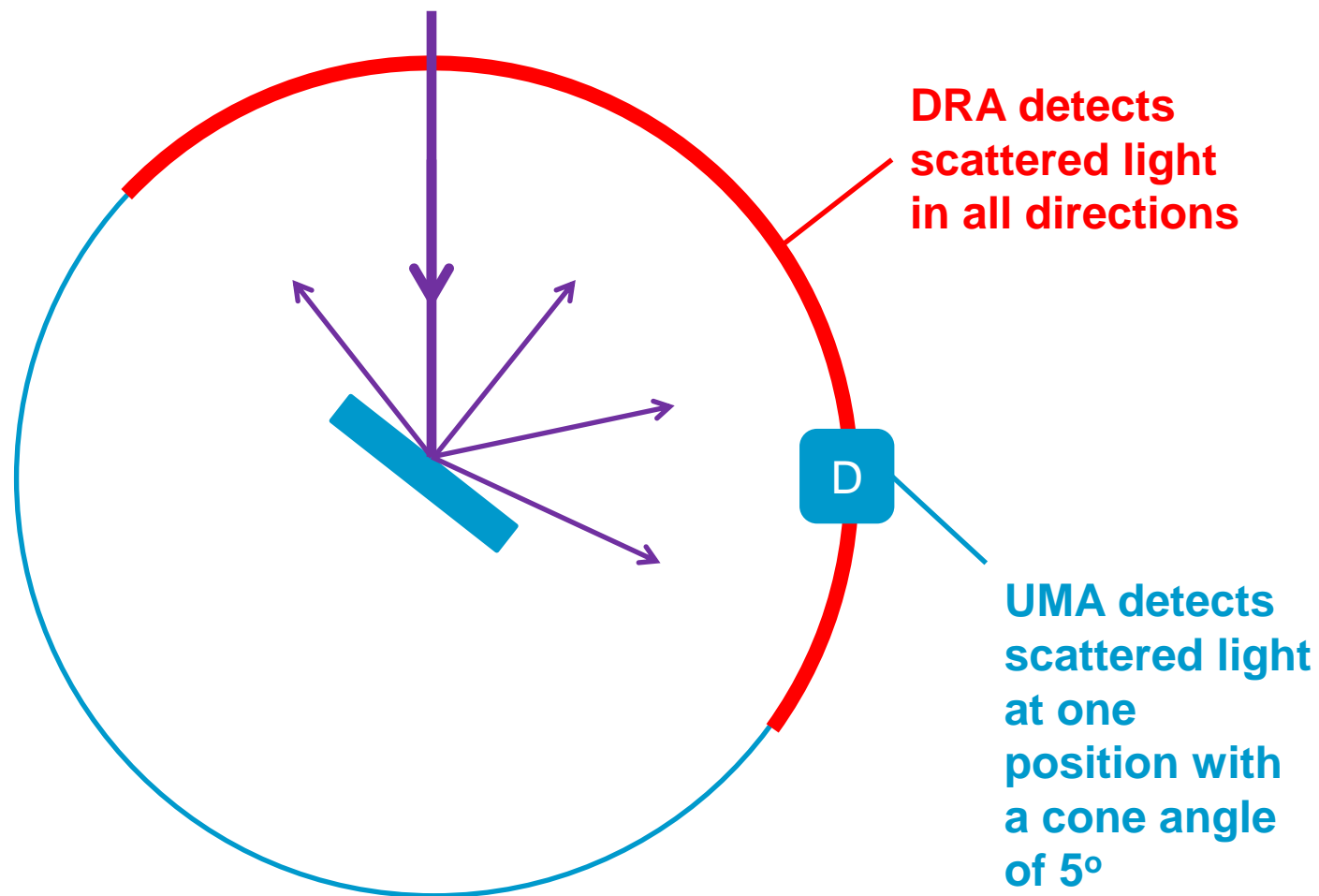
Red data interval = 1 nm   Black data interval = 10 nm

# Scattered Reflection

Lambertian - PTFE



# UMA vs DRA



# Diffuse Colour Standards



# BLUE PTFE COLOUR STANDARD

## UMA, Internal DRA, External DRA

### All Accessories:

- Double Beam Auto Select
- UV/Vis
  - Data Interval = 1 nm
  - SBW = 4 nm
- NIR
  - Data Interval = 1 nm
  - Energy = 3

### UMA:

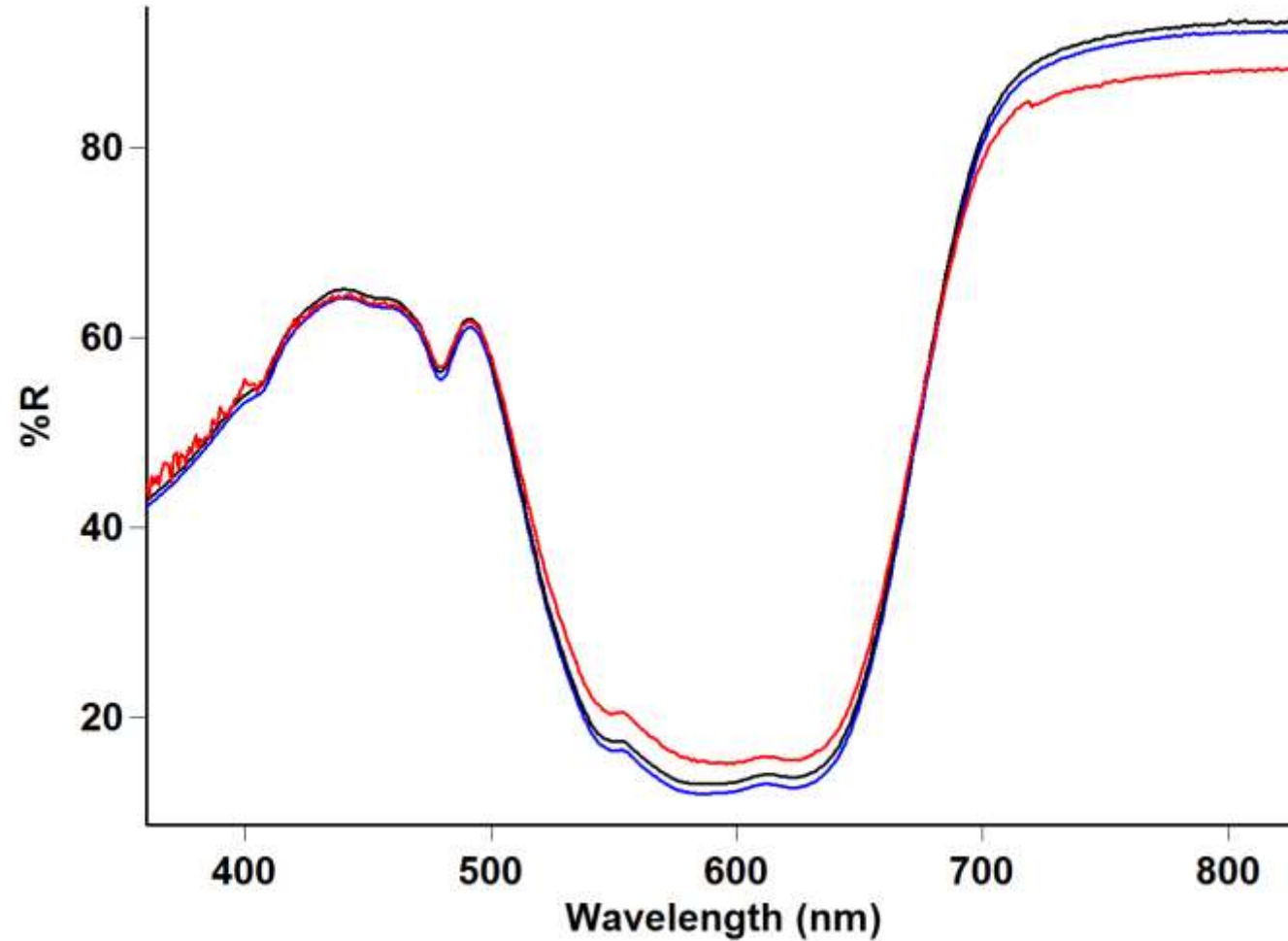
- SAT = 0.5 s

### Internal DRA

- SAT = 0.1 s

### External DRA

- SAT = 0.1 s





**The End**  
**Thank You for Your Patience**

**Questions?**

Contact Information :  
[mark.fisher@agilent.com](mailto:mark.fisher@agilent.com)